DESIGN REPORT FOR THE ATTACHMENT Z-1 REMEDY ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, INDIANA

REVISION: 1

Prepared for

Enviro-Chem Site Trust Fund

Submitted to

United States Environmental Protection Agency, Region 5
and
Indiana Department of Environmental Management

Submitted by

ENVIRON International Corporation
Deerfield, Illinois

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(continued)

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ACRONYMS AND ABBREVIATIONS

ARARs	applicable relevant and appropriate requirements
cfm	cubic feet per minute
cis-1,2-DCE	cis-1,2-dichloroethene
COCs	contaminants of concern
°F	degrees Fahrenheit
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
ENVIRON	ENVIRON International Corporation
ESD	Explanation of Significant Differences
FSP	Field Sampling Plan
GAC	granular activated carbon
gpm	gallons per minute
HDPE	high-density polyethylene
IDEM	Indiana Department of Environmental Management
mg/L	milligrams per liter
PID	photoionization detector
PLC	Program Logic Controller
PRGS	permeable reactive gate system
PVC	polyvinyl chloride
QAPP	Quality Assurance Project Plan
ROD	Record of Decision
scfm	standard cubic feet per minute
Site	Enviro-Chem Corporation Superfund Site
SPLP	Synthetic Precipitate Leaching Procedure
SVE	soil vapor extraction
SVOCs	semivolatile organic compounds
TBCW	thin barrier wall curtain
Trust	ECC Site Trust
TSS	total suspended solids
USEPA	United States Environmental Protection Agency
VOCs	volatile organic compounds

1.0 INTRODUCTION

1.1 Introduction

This Design Report for the Attachment Z-1 Remedy (the "Design Report") has been prepared for the Enviro-Chem Superfund Site ("ECC Site" or the "Site"), located in Zionsville, Indiana. It is submitted by ENVIRON International Corporation (ENVIRON) on behalf of the ECC Site Trust (the "Trust").

1.2 Background

As presently configured, the SVE system that has been installed at the ECC Site has not achieved the subsurface water cleanup standards in the till, which are set forth in Table 3-1 to Revised Exhibit A. The United States Environmental Protection Agency (USEPA) and Indiana Department of Environmental Management (IDEM) are concerned that failure to achieve those cleanup standards may, over time, have an adverse effect on water quality in Unnamed Ditch, which is located adjacent to the eastern portion of the Site. For that reason, Revised Exhibit A, the Consent Decree, and the amended Record of Decision[1] (ROD) provide for specific Additional Work to be performed if USEPA determines that those standards were not met within a 5-year period, unless the parties agree otherwise.

These standards were not met within the 5-year period provided in the Consent Decree. The agreed modifications to the "Additional Work" provisions of Revised Exhibit A and the Consent Decree were presented in Attachment Z-1. The Attachment Z-1 Remedy includes an augmented soil vapor extraction (SVE) system that augments the existing SVE system by installing additional SVE trenches generally along the alignment of a ground water collection trench previously required as Additional Work in Revised Exhibit A to the Consent Decree. The new SVE trenches will be connected to the existing SVE system and will be operated using all of the basic operations of the existing SVE system equipment. In order to provide additional protection to Unnamed Ditch, the Attachment Z-1 Remedy also includes a perimeter thin barrier curtain wall (TBCW), which was constructed in May 2006, and a permeable reactive gate system (PRGS). The Attachment Z-1 Remedy enhances and replaces the water interception trench originally required as the Additional Work in Revised Exhibit A. The Attachment Z-1 work will be conducted under the Additional Work provisions of the Consent Decree, as amended.

After construction of the augmented SVE trenches and the PRGS is completed, there will be several distinct phases for the operation of the modified Additional Work. The activities will be different for each period. The periods and the associated activities are as follows:

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[1] The original ROD for the Site was issued in September 1987, and the Amended ROD was issued in June 1991.

- **Active Phase**. This is defined as the period of operation of the augmented SVE trench system.

- **Phase I Monitoring**. This is defined as the 1-year period beginning when the Soil Vapor Standards have been achieved in the augmented SVE trenches. At the completion of the Phase I Monitoring period, Phase II Long-Term Monitoring will begin at the Site.

- **Phase II Long-Term Monitoring**. This is defined as the period following the completion of Phase I Monitoring. Phase II Long-Term Monitoring is divided into Phase II(a) and Phase II(b), as noted below.

2.0 ATTACHMENT Z-1 REMEDY DESIGN

2.1 Attachment Z-1 Remedy Activities

The TBCW, piezometer installation, and till water pump test portions of the Attachment Z-1 Remedy have been completed. The TBCW was installed along the east, south, and southwest sides of the Site in May 2006. Four sets of three piezometers were installed in June 2006 along the length of the TBCW. For each set of piezometers, one piezometer was installed in the till unit downgradient of the TBCW, a second piezometer was installed in the till unit upgradient of the TBCW, and a third piezometer was installed within the sand and gravel unit, adjacent to the upgradient till unit piezometer.

The till water pump test was completed in July 2006. The *Till Water Pump Testing Report*, which contained descriptions of the performance test methodologies, test observations, and results, was submitted in August 2006. This report concluded that there is no hydraulic connection between the till unit and the underlying sand and gravel unit in the northern and central portions of the Site (based on the results from well T-1), on the south and southwest sides of the Site (based on the results from well HS-1), and in the southeast corner of the Site (based on the results from well HS-2). Accordingly, the results of the till water extraction tests indicate that it will not be necessary to use vertical SVE wells in place of Trench Segments 5 and 6; rather, SVE trenches can be installed and operated across the entire alignment of the proposed augmented SVE trench system.

Technical Specifications for the major construction components of the Attachment Z-1 Remedy are included in Appendix A. The sequence of activities for implementing the remaining tasks of the Attachment Z-1 Remedy is presented below.

- Installation of SVE trenches along the east, south, and southwest sides of the Site.
- Installation of the PRGS.
- Monitoring of surface and subsurface water for compliance with Site-Specific Acceptable Concentrations (Table 2-1).
- Collection and treatment of subsurface and till water and soil vapors via the augmented SVE trench system until attainment of the Soil Vapor Standards listed in Table Z-1-3 of Attachment Z-1 (incorporated herein as Table 2-2).
- After SVE operation, treatment of trench system till water using the PRGS.

2.2 Site Preparation and Site Controls

2.2.1 Support Zone Facilities

Prior to installation of the remaining Attachment Z-1 Remedy components, the Contractor will establish an on-site Support Zone for "clean" operations. The location of the Support Zone is shown on Drawing C-2. The Support Zone will contain temporary Site facilities, including an office trailer, toilet facilities, vehicle parking areas, utility hookups, equipment staging areas, and container staging areas for potentially contaminated materials. An office trailer, some utility hookups, vehicle parking areas, an access road, and a decontamination pad currently exist at the Site. If necessary, designated parking areas will also be located along the access road to the Northside Landfill. Locations for certain Support Zone facilities are depicted on Drawing C-2; however, the Support Zone facility locations may be adjusted by the Contractor, with the approval of the Trust's Engineer.

2.2.2 Erosion and Sedimentation Control Measures

During construction, soil erosion and sedimentation control measures will be undertaken, in accordance with Indiana Best Management Practices,[2] between all potential sources of exposed erodable soils and the nearest surface water (e.g., Unnamed Ditch or ECC drainage ditches). Existing silt fencing may be utilized if it is repaired and accumulated soils are removed. Sediments trapped behind silt fences will be excavated during and after completion of construction and spread in areas that are not subject to erosion prior to restoration of those areas (e.g., placement of topsoil, seeding, etc.).

Visual assessments will be performed during construction activities to determine the need for dust control. Typical dust control methods include water application and modifications to hauling routes.

2.2.3 Site Access Restrictions

Access restrictions will be implemented during the construction activities and during the Active Phase and Phase I Monitoring. Thereafter, access restrictions may be modified, if USEPA agrees, as provided in the USEPA June 2006 Explanation of Significant Differences (ESD). All personnel and visitors will be required to sign in and out at the main office of Boone County Resource Recovery or the project construction trailer. No visitors without proper protective equipment and training will be allowed on site during intrusive construction activities.

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[2] See "Indiana Handbook for Erosion Control in Developing Areas" and/or 327 IAC 15 Rule 5 for guidance regarding Best Management Practices.

Access restrictions will include the maintenance of an 8-foot high chain-link fence around the perimeter of the Site. The eastern portion of this fence has been temporarily removed for the construction period. However, the adjacent Northside Landfill fence will provide adequate protection during this time. The eastern fence will be replaced at the beginning of the Active Phase.

2.3 Augmented SVE System Overview

The augmented SVE system will be used for SVE treatment of the shallow till along the east, south, and southwest sides of the Site. The existing SVE system will be augmented by additional trenches, which will be connected to the existing SVE system and will be operated using the nine basic operations of the existing SVE system. The nine basic operations are as follows:

1. Aeration and equalization of "raw water" collected from dewatering of the trenches.

2. Transfer of the water to the Treatment Building using influent feed pumps.

3. Filtration of the influent water using total suspended solids (TSS) filters.

4. Water treatment using a counter-current tray aeration air stripper.

5. Combining the air stripper "off-gas" with the SVE "air header pipe."

6. Absorption of organics in the combined air stream using granular activated carbon prior to release to the atmosphere.

7. Filtration of air stripper effluent water through additional TSS filters.

8. Absorption of residual organics in the filtered air stripper effluent water using granular activated carbon.

9. Discharge of treated water to Unnamed Ditch.

Contaminated moisture in the sand lenses is likely to be the principal mechanism by which contamination is transmitted to the trenches. The SVE system is expected to remove that moisture. Free liquid entrained in the vapor removed by the SVE system will be drawn to the Treatment Building and will there be removed by gravity in an entrainment separator. Periodically, water that accumulates in the entrainment separator will be pumped to an on-site ECC storage tank for

subsequent treatment in the on-site Treatment Building and discharged in accordance with the substantive requirements of applicable federal and state laws, and meet the effluent limits included in Table 2-3.

Existing vacuum pumps will be used for the collection of contaminants via soil vapors. The collected soil vapors will pass through a new carbon adsorption system, which will consist of two carbon vessels connected in series, as described in Section 2.6.

The augmented SVE system adds seven segments (i.e., Segments 1 through 7) to the existing SVE trench layout, each of varying length. The locations of the augmented SVE trench segments are shown on Drawing C-3. The trenches are situated to intercept permeable lenses in the till unit, above the sand and gravel unit (see Drawing C-4). Trench segment coordinates are provided in the Design Drawings C-3 through C-8. A short lateral from the south end of SVE Trench Segment 5 extends into Hot Spot Area 2, and a short lateral from the western portion of Trench Segment 6 extends into Hot Spot Areas 1 and 1A.

SVE Trench Segments 4 and 5 are located along the approximate alignment of a sheet pile wall section that was installed as part of the 1998 Site work. The metal sheeting may be removed or cut off, as needed, to allow excavation of the trench segments as shown on Drawing C-3.

2.4 Augmented SVE Trenches

2.4.1 SVE Trench Design/Construction

Each of the augmented SVE trenches will be approximately 24 inches wide. The trench depths are depicted on Drawings C-4 through C-8 and listed in Schedule C on Drawing C-10. The SVE trench excavations will be performed through biopolymer slurry, such as natural or synthetic guar gum, to prevent the trench walls from collapsing during the excavation and to reduce the potential for heaving of the lower sand and gravel unit at the bottom of the excavation. The Contractor is responsible for supply and storage of water used for the biopolymer slurry mix. Soils excavated from the upper 2 feet of the trench, as well as the widened upper portion of the trench discussed below, should be stockpiled separately for reuse as surface backfill. Prior to reuse, the stockpiles will be screened using a photoionization detector and significantly impacted soils will be handled as described in Section 2.8.

Excavation to exactly the specified depth is critical. Excavation beyond the design depth may result in the interception of the lower sand and gravel unit, which will adversely affect the dewatering of the trench. Excavation that is more than 6 inches to 1 foot too shallow could adversely affect the passive water drainage system to the PRGS. Biopolymer slurry will be added to the trenches, as necessary, as the excavations proceed to maintain the level of biopolymer slurry in the trench to within approximately 2 feet of the ground surface.

After the excavation depth has been verified in each trench segment, the trench segments will be backfilled with free-draining, 1- to 2-inch diameter gravel. While backfilling is performed, one vertical 4-inch diameter polyvinyl chloride (PVC) riser pipe and slotted well screen will be installed within each trench segment for trench dewatering. These dewatering wells will be installed near the low point of each trench segment and will be used for initial development/biopolymer slurry removal (see discussion below), as well as initial and possibly periodic dewatering of the augmented SVE trenches. Each dewatering well will be equipped with a 10-foot vertical section of slotted PVC well screen at its base, as appropriate, depending on the total depth of the trench at the respective location of the dewatering well. Solid PVC casing will extend from the well screen to the surface (see Drawings C-5 through C-8 and the detail on Drawing C-10). The Contractor may install additional temporary vertical PVC piping for the addition of enzymes to dissolve the biopolymer slurry after completing each trench, if desired.

As each trench is backfilled, horizontal 4-inch diameter solid PVC PRGS conveyance piping and a slotted 4-inch diameter horizontal PVC pipe (SVE screen) will be installed. The SVE/PRGS piping in Trench Segments 1 through 7 is shown on Drawings C-5 through C-8.

The PRGS conveyance piping placement has been designed to transfer by gravity the water collected in the trench segments during the Phase I Monitoring and Phase II Long-Term Monitoring periods to a PRGS collection manhole. In order to ensure proper flow to the collection manhole, the depth at which each pipe segment is installed is also critical and must conform exactly to the elevations shown on Drawings C-5 through C-8 and listed on Schedule B on Drawing C-10. Discharge piping will be installed to carry the PRGS treatment vessel effluent to Unnamed Ditch.

When the trench backfill reaches the design elevation for the PRGS piping, the PRGS conveyance piping will be installed through the biopolymer slurry. The design elevations for the PRGS piping is shown on Drawings C-5 though C-8 and are listed on Schedule B on Drawing C-10. At the Contractor's discretion, if artesian conditions are not present, the level of the biopolymer slurry may be pumped down to the level of the top of the gravel backfill during pipe installation. When the end of the SVE trench segment is reached, the PRGS conveyance piping will continue beyond the SVE trench segment, in a separate pipe trench, to a connection manhole where a T connection, gate valve, and one-way valve will be installed. The PRGS conveyance piping will then continue into the next trench segment, if necessary.

When the trench backfill reaches the design elevation of the SVE piping, the SVE screen will be installed horizontally at the design elevation, which is shown on Drawings

C-5 through C-8 and listed on Schedule A on Drawing C-10.[3] The SVE screen will collect soil vapor during the Active Phase and will collect subsurface water during the Phase I Monitoring and Phase II Long-Term Monitoring periods. The placement elevation for the SVE screen and outflow connection to the PRGS conveyance pipe will control the potentiometric surface of the till water along the eastern and southern boundaries of the Site during the monitoring phases, thereby preventing the buildup of till water on the upgradient side of the TBCW.

The SVE screen will connect to a 10-foot section of non-slotted PVC pipe near the end of the trench segment. The non-slotted section will continue beyond the SVE trench segment, in a separate pipe trench, to the adjacent connection manhole. The SVE pipe terminates at the manhole, where a T connection will connect it to the PRGS conveyance piping through the gate valve and a one-way valve. The gate valve will be closed during the Active Phase and opened at the end of the Active Phase.

The SVE screen will be fabricated with solid vertical PVC access pipes installed at two locations on each of the seven trenches and extending to ground surface. One of these vertical PVC pipes (4-inch diameter) will be installed at the end of the SVE trench segment for instrumentation monitoring and will be equipped with a glycol-filled vacuum gage (pressure range to be determined in the field). The other vertical pipe (4-inch diameter) will be installed near the center of each of the trench segments and will be connected at the ground surface to piping from the existing SVE vacuum blower system. This vacuum inlet pipe will include an adjustable valve, a flow measurement port, sampling port, and vacuum gage.

After the dewatering wells, PRGS conveyance piping, SVE piping, and access pipes are installed, the gravel backfill will be placed in the trench to 2 feet below the ground surface. An appropriate enzyme will then be added by the Contractor to dissolve the biopolymer. The trench water containing the enzyme and dissolved biopolymer will be sampled and tested, as described in the Technical Specification (Appendix A) and the Addendum to the Field Sampling Plan (FSP) in Appendix C. When the biopolymer has been sufficiently dissolved, it will be pumped out of the trenches using the 4-inch diameter dewatering wells and sent to the ECC wastewater treatment system.[4] Any biopolymer solution displaced during the construction activities or removed from the PVC riser pipes will also be treated with enzyme and sent to the ECC wastewater treatment system. The wastewater treatment system will manage the treated biopolymer solution in accordance with applicable standards and applicable relevant and appropriate requirements (ARARs).

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[3] SVE pipe elevation within 0.2 feet of the design elevation is critical to the water collection function of this piping and must be maintained.

[4] Additional temporary wells may be installed as needed by the Contractor during backfilling to be used for flushing the biopolymer slurry.

2.4.2 Augmented SVE Trench Performance Test

An initial attempt to dewater the augmented SVE trench segments will occur immediately after the removal of the dissolved biopolymer slurry. Difficulty dewatering the trench segments using the maximum anticipated dewatering rate will be an indicator that the trench has intercepted the underlying sand and gravel unit, in which case the Contractor may be required to grout the sand zone and/or reinstall the trench segment at a shallower depth. Assuming that the sand and gravel layer has not been intercepted, the maximum anticipated dewatering rate for individual trench segment is expected to be less than 5 gallons per minute (gpm).

2.4.3 Augmented SVE Trench Completion

After initial dewatering/testing (see Section 2.4.2 above), the SVE trench segment will be widened approximately 1 foot on each side of the SVE trench, for a total width of approximately 4 feet, from the ground surface to 2 feet below the surface to allow for the installation of a seal that will minimize vacuum leaks from the ground surface into the SVE trenches. A geomembrane underlain by a non-woven geotextile will be installed approximately 2 feet below ground surface over the gravel backfill in each trench and the widened section. The geomembrane may be either high-density polyethylene (HDPE) or approved, laminated, scrim-reinforced sheeting. A patch will be used to prevent vacuum leaks at the penetrations for the dewatering well and other riser pipes. The patches will be secured to each well and riser pipe using hose-claps and silicon sealant or manufacturer's recommended methods. The configuration and dimensions for the geomembrane seal are shown on Drawing C-10. The soil excavated from the upper 2 feet may be replaced as compacted backfill in the top 2 feet of the trench segment if it is not contaminated based on observations or photoionization detector measurements and it meets the requirements for common fill (Appendix A). The soil above the geomembrane will be compacted in at least two lifts.

2.5 Augmented SVE Piping and Connections

2.5.1 SVE Screen Connections

The SVE screen and the 4-inch PVC vacuum inlet riser will be connected at the ground surface to 4-inch diameter piping that runs to the ECC Treatment Building (Drawing C-9). This 4-inch SVE piping will be fitted to the existing 3-inch diameter SVE manifold, (which currently connects to the vacuum/blower) using flexible pipe and camlock clamps (Drawing C-9). Condensate traps with drains will be included in the new piping alignment to the Treatment Building and will be drained, as needed, by the Contractor.

In the PRGS manhole, the SVE piping will be connected to the PRGS piping via a gate valve and top operating nut, such as an ASHAI/America GT-B model. This valve will be closed initially, but will allow the connection to the PRGS conveyance piping to be opened when SVE activities (i.e., the Active Phase) are complete.

2.5.2 PRGS Piping Connections

As noted above, a T will be incorporated into each horizontal SVE screen, which will connect the SVE pipe to the PRGS conveyance pipe in the manhole beyond each SVE trench segment. A one-way check valve will be installed between the two pipes. A gate valve to be installed just before the T, will be closed during the Active Phase and opened during Phase I Monitoring and Phase II Long-Term Monitoring Phases.

Another T connection and valve will be installed in line with the PRGS conveyance piping at each manhole. The valve will control the PRGS pipe connection between trench segments. This valve will be closed during the Active Phase and opened during the Phase I Monitoring and Phase II Long-Term Monitoring periods. When the SVE pipe gate valves and the PRGS pipe valves are opened, ground water accumulating in the SVE trenches above the SVE screen elevation will be conveyed by gravity south to the PRGS collection manhole. This water will be pumped to the treatment vessel.

The other end of the PRGS pipe T section will be capped for future clean out of the piping, as needed.

2.5.3 Dewatering Piping

The dewatering well in each of the trench segments will be fitted with a submersible pump with 1.25-inch PVC piping leading to 0.75-inch diameter PVC piping. A Grundfos 10-Rediflow-3-100, or approved equivalent pump, shall be used in each well.[5] The 1.25-inch piping will connect to 0.75-inch piping near the ground surface and run along the ground to one of two temporary aboveground water tanks (500-gallon capacity). Heat tracing of the dewatering piping and tank heaters are included in the design for freeze protection.[6] The 0.75-horsepower submersible pumps installed in each temporary aboveground water tank will send the collected water to a new 150,000-gallon water storage tank (Tank T-5) to be added near the Treatment Building. Piping from the temporary water tanks (Tanks T-6 and T-7) will be 1.5-inch diameter PVC. Tank T-5 will be connected to the existing piping for transfer to the Treatment Building. Dewatering piping is shown on Drawing C-11.

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[5] The pump will not be installed unless measurable water accumulates in the dewatering well.

[6] The dewatering is expected to be temporary and performed during non-freezing conditions.

2.5.4 Electrical Connections

The dewatering well pumps (i.e., 1/3-horsepower, single-phase, 115-volt pumps), dewatering well pump control switches, transfer pumps in each temporary water tank, pump control switches in each temporary tank, and the pump/diffuser in the new 150,000-gallon storage tank all will require power to operate. Conduits will run from the electrical panel(s) at the Treatment Building to junction boxes installed at each dewatering well. Instrumentation (switch) cables will be separate from the power supply lines. The addition of an electrical panel is anticipated. Drawings E-1 and E-2 provide the general electrical requirements. Electrical system details are to be determined by the Contractor to the extent not provided in the design and in accordance with local codes.

2.6 Soil Vapor and Water Treatment System Design/Upgrade

The existing ECC treatment facility will be used for the treatment of the contaminated subsurface water and soil gas collected during the dewatering efforts and Active Phase. As shown on Drawings C-11 and P-1, the subsurface water will be pumped from the augmented SVE trenches to two temporary tanks (T-6 and T-7), then to Tank T-5 before being conveyed to the existing ECC Treatment Building, via existing underground piping and existing centrifugal transfer pumps. Tank T-5 will be equipped with a differential pressure transmitter or approved switches to control the centrifugal transfer pumps (pump on, pump off, critical high-level, and critical low-level). The existing Program Logic Controller (PLC) in the ECC Treatment Building will be upgraded to provide connections to shut down the pumps in the augmented SVE trenches if critical levels are reached in the storage tanks or any of the treatment system transfer pumps shut down.

The existing SVE system includes two 60-horsepower vacuum pumps, each with a design vacuum of 10 inches of mercury and a design backpressure of 1.25 inches of mercury. The design airflow is 1,175 cubic feet per minute (cfm) per blower and the design discharge temperature is less than 175 degrees Fahrenheit (°F). Prior to treatment, the vapor is cooled to increase effectiveness of the carbon units. The heat exchanger has a normal airflow of 2,400 cfm and a normal water flow of 0.75 to 7 gpm.

A 1998 performance test of the vacuum pumps showed a flow rate for each blower of 1,290 standard cubic feet per minute (scfm) with a vacuum of 10 inches of mercury.[7] The existing SVE system has approximately 4,300 feet of trenches, which is equivalent to 0.6 scfm per foot of trench with both blowers operational. The augmented SVE system will have approximately 975 feet of trenches, which is equivalent to 1.32 scfm per foot of trench with a single blower operational. Even with the addition of an *ex situ* SVE treatment cell (maximum 300 feet of

[7] Documented by Versar in the *Draft Soil Vapor Extraction System Start-Up Report for the Enviro-Chem Superfund Site, Zionsville, Indiana*, dated May 1999.

additional SVE line); one of the existing blowers is expected to be sufficient to power the augmented SVE system.

For this augmented SVE system design, the source of contaminants to be extracted is associated with till water and the soils in contact with the granular lenses. The system will allow for the collection of vapor samples from the individual SVE trenches or the combined air stream. Also, the extracted vapors can be monitored continuously by in-line instrumentation, including a photoionization detector (PID) and moisture analyzer. The on-line analyses will be used for screening and operations optimization.

The SVE system will be capable of removing moisture along with the soil vapor that accumulates in the SVE trenches. Free liquid in the extracted vapor will be separated by gravity in an entrainment separator in the Treatment Building. A level control system will be utilized to control the removal of water that accumulates in the entrainment separator as required. The existing separator tank is equipped with a vacuum breaker system, which will open the tank to the atmosphere to permit water to be transferred by pump from the separator to Tank T-5, as necessary.

The exhaust from the soil vapor vacuum pump system is connected to a two-stage carbon adsorption system (i.e., primary and secondary). This system consists of two vessels in series containing granular activated carbon (GAC) that will be used by the adjacent Third Site. New GAC vessels will be added for treatment of ECC wastewater. The organics contained in the extracted air will be adsorbed on the activated carbon. The moisture content of the air stream will be less than 50% relative humidity, and temperatures will be maintained below 150 °F by a cooling system. These conditions allow for efficient operation of the carbon adsorption unit.

The vapor from the primary carbon vessel will be monitored frequently by an existing on-line organic analyzer. When the organic analyzer detects organic vapor in the air stream between the primary and secondary carbon vessels, the SVE system will shut down automatically to permit the removal and replacement of the "spent" primary carbon vessel. An operator will be alerted to this condition by the shutdown alarm, and will disconnect the primary carbon vessel from service. The spent carbon vessel will be removed and a carbon vessel containing fresh activated carbon will be placed in operation. The unit previously serving as the secondary carbon bed will become the primary carbon bed and the unit just placed in operation will be the secondary carbon bed. Once this switch is complete, the SVE system (i.e., vacuum pump and injection pump) will be restarted and the system operation resumed. The arrangement of two activated carbon vessels in series (i.e., primary and secondary) will permit optimal utilization of the activated carbon, and efficient capture of the organics. The spent carbon vessels will be temporarily stored on site. The inlet and outlet connections to each carbon vessel will be capped and sealed appropriately. Periodically when a truckload quantity of vessels has accumulated, and at the conclusion of the Active Phase, the vessels containing the spent carbon will be transported in accordance with the requirements of the applicable federal and state laws and ARARs to an off-site facility where the carbon will be

regenerated by high temperature incineration, and in the process, the organics adsorbed on the carbon will be destroyed.

Wastewater discharges and vapor emissions from the existing treatment systems will be monitored to ensure attainment of the standards presented in IDEM's February 1997 *Briefing Memorandum on ARAR Effluent Limits.*

Additional details concerning the ECC treatment facility and its operation are presented in the following Site documents:

- *100% SVE & WWT Design for the RRA at the Enviro-Chem Superfund Site, Zionsville Indiana*, Versar and Handex, November 14, 1997.

- *Operation and Maintenance Manual-Soil Vapor Extraction and Ground Water Remediation Systems Enviro-Chem Superfund Project*, Handex of Indiana, January 1999.

- *Revised Remedial Action (Exhibit A) – Revision 2*, May 7, 1997.

Prior to start of the Active Phase, the following construction activities at the ECC treatment facility are anticipated:

- Construction/installation of new wastewater tank T-5.
- Installation of two dedicated air and two dedicated water GAC units.
- Upgrade of existing electrical panel and addition of a new panel.
- Installation of additional program/connection capability in the existing PLC.
- Reprogramming of the existing teleadialer to access the existing PLC. A summary of the required alarm conditions is provided in Table 2-4.

The Contractor will be responsible for performing all necessary ECC treatment facility maintenance and modifications to allow for treatment of the Site ground water and for efficient operation and monitoring of the facility. Soil vapor sampling, will be conducted by the Contractor during system operation. Vapor sampling to demonstrate achievement of the required vapor standards will be performed by the Trust's Engineer with cooperation of the Contractor.

2.7 Permeable Reactive Gate System

2.7.1 PRGS Description

The PRGS will be installed concurrent with the SVE trench construction, but will not be activated until the Soil Vapor Standards have been achieved at the end of the Active Phase. The PRGS is intended to act as a backup system that will prevent build-up of till water on the upgradient side of the TBCW and will treat till water that is allowed to leave this area during the monitoring phases. Due to the presence of the cap on the northern portion of the Site, very little till water is expected to build up along the upgradient side of the adjacent TBCW.

The PRGS treatment vessel is a gravity-driven, flow-through treatment system that will utilize zero-valent granular iron to degrade chlorinated compounds to inorganic chloride and dehalogenated organic compounds. This treatment technology has been demonstrated during numerous bench scale studies, pilot studies, and full-scale remediation projects for various chlorinated compounds in ground water and wastewater. Based on recent research, the predominant degradation pathways are expected to consist of: (1) oxidation of the iron due to the presence of dissolved oxygen (DO) in ground water entering the treatment system creating hydroxyl radicals that degrade the chlorinated compounds and (2) further degradation of chlorinated compounds during reducing reactions with iron, a strong reducing agent. The chlorinated compounds are degraded to inorganic chloride, ethene and ethane, partially dechlorinated byproducts (e.g., 1,2-dichloroethene; 1,1 dichloroethene; and vinyl chloride) and small-chained hydrocarbons (e.g., methane and propane). These reactions are accompanied by the hydrolysis of water and subsequent formation of hydrogen gas. The partially dechlorinated by-products can be treated further by the same reducing reactions with iron given sufficient retention time in the PRGS.

A PRGS treatment vessel has been designed to maintain appropriate flow velocities through the vessel, thereby, allowing for appropriate detention time for treatment of the till water. A vent in the vessel hatch is included to avoid build-up of pressures from the formation of hydrogen gas during the reactive process. Design details are presented in Appendix D. As discussed in Appendix D, cis-1,2-dichloroethene (cis-1,2-DCE) is the constituent of concern currently present in the till water requiring the longest detention time based on published information on degradation rates in PRGSs and maximum concentrations historically detected in subsurface water samples collected from till monitoring wells. As such, the PRGS is designed to treat collected ground water to concentrations below the Discharge Limits provided in Table 2-3, specifically to treat cis-1,2-DCE to below the direct discharge limit of 0.002 milligrams per liter (mg/L) established by IDEM on February 27, 1997.

2.7.2 PRGS Design/Construction

The PRGS will utilize the SVE screen to collect till water, which will then be conveyed in separate PRGS piping by gravity to a collection manhole and then pumped to a treatment vessel. The PRGS conveyance piping will be installed in the SVE trenches during trench construction. The connections between the SVE screen and the PRGS conveyance piping in the manholes adjacent to the trenches are shown on Drawing C-10.

PRGS conveyance pipes from the east and west sides of the Site will terminate in a PRGS collection manhole located north of the PRGS treatment vessel. The manhole will be waterproofed and equipped with a pump to move the water that accumulates in the manhole to the PRGS treatment vessel. A pump removed from one of the dewatering wells can be used. One 1.25- to 1.5-inch diameter pipe will extend from the pump in the PRGS collection manhole to the treatment vessel inlet.

The PRGS treatment vessel will be installed at the south end of the Site, north of the TBCW. The vessel will consist of a fabricated concrete tank, which will be fitted with holes for the influent and effluent pipes, an access/sample collection pipe, and a water-tight vented hatch door. A pretreatment zone containing a mixture of granular iron and sand will be placed at the influent (north) end of the vessel. A treatment zone containing 100% granular iron will be placed in the middle section of the vessel. The south end of the vessel contains piping to transfer PRGS vessel effluent to the discharge piping. The effluent pipe will be fitted with an access/sample port. The vessel shall include adequate venting (to prevent the buildup of hydrogen gas), water proofing, and adequate structural support.

The primary purpose of the sand/iron pretreatment zone is to remove dissolved oxygen present in the influent water, thereby prolonging the effective life of the granular iron treatment bed. The sand/iron mixture will contain 10% iron and 90% sand by weight.

Influent and effluent piping will be installed at the ends of the treatment vessel. Perforated piping wrapped with a geotextile and surrounded by a 6-inch deep sand bed will be installed at the base of the north and middle sections and will be used to collect water to transfer to the next vessel section. The collection pipe outlet in the middle section will extend to a height of at least 1 foot below the inlet pipe in the north section to maintain a saturated treatment section and flow.

The treated (effluent) water will flow by gravity out of the treatment vessel through an outflow pipe that extends through SVE Trench Segment 6 and the upper zone of the existing TBCW to Unnamed Ditch. The discharge pipe will be fitted with a valve to prevent backflow to the reservoir in the event of surface water flooding of the area outside the TBCW.

The PRGS location and design details are presented on Drawing C-12 and in Section 2206 of the Technical Specifications. Design calculations used to determine the size and configuration of the PRGS are presented in Appendix D.

2.8 Waste Management

2.8.1 Excavated Soil Management

After removal and segregated staging of the upper 2 feet of soil from the upper widened portion of each augmented SVE trench, excess spoils from excavation of the new SVE trench segments will be temporarily placed in staging areas adjacent to the trenches so that the biopolymer slurry will drain from the spoils and flow back into the excavation. Berms and/or silt fencing will be maintained along the Unnamed Ditch to prevent biopolymer solids or excavation spoils from entering the Unnamed Ditch.

Figure 2-1 is a decision tree for the management of the excess excavated trench soils. At least one sample from each stockpile of the excavated soil from the augmented SVE trench excavation will be obtained and tested as specified in the Addendum to the FSP (Appendix C). Each stockpile of soil (at least one per trench segment) shall be managed separately according to the decision tree. If the stockpile of soil exceeds the Site-Specific Soil Exposure Concentrations as listed in Table Z-1-2 in Attachment Z-1 (incorporated as Table 2-5 in this Design Report)[8] or the Synthetic Precipitate Leaching Procedure (SPLP) test results exceed the Acceptable Stream Concentrations listed in Table 2-1 of this Design Report, the excavated soils will be handled as follows:

- If the soil exceeds the Site-Specific Soil Exposure Concentrations, then the soil will be treated on site using an *ex situ* SVE procedure to achieve the Site-Specific Soil Exposure Concentrations. The *ex situ* SVE will occur in a treatment cell constructed on the Northern or Central SVE Treatment Area. The *ex situ* SVE soil treatment cell will utilize the existing SVE treatment system (blowers, air GAC) and the *ex situ* SVE will be conducted using the same procedures as trench segments. The design for the *ex situ* SVE treatment cell is presented on Drawing C-13; however, the size will be adjusted based on the volume of soils to be treated.

[8] The Acceptable Soil Concentrations reflect adjustments from Table 3-1 of the Consent Decree based on consideration of potential human exposure pathways at the Site as provided for in Footnote 4 of Attachment Z-1, Table Z-1-1 and Attachment Z-1, Table Z-1-2.

- If the soil does not exceed the Site-Specific Soil Exposure Concentrations as listed in Table 2-5 (either before or after on-site treatment, if any) it will be analyzed using SPLP methods. If the SPLP of the soil exceeds the Applicable Stream Concentrations as listed in Table 2-1, the soil will either be disposed off site according to applicable USEPA and IDEM regulations and ARARs or placed beneath a 12-inch minimum vegetated soil cover of uncontaminated soils on the Northern or Central SVE Treatment Areas.

- If the soil does not exceed the Site-Specific Soil Exposure Concentrations and the SPLP analyses do not exceed the Applicable Stream Concentrations, then the soil will be placed on the Northern or Central portions of the Site and vegetated.

Since the actual volume of soil to be treated will be determined during the construction phase, a contractor submittal will be provided with *ex situ* cell details at that time. The calculations for the anticipated soil excavation volume are contained in Table B-2 of Appendix B. If needed, the *ex situ* treatment cell will contain up to three 4-inch diameter slotted PVC pipes connected to non-slotted (solid) 4-inch diameter PVC piping approximately 5 feet in from the edge of the treatment cell. Solid 4-inch PVC piping will extend out of the cell and through the wall of the existing Treatment Building. This piping will be fitted with camlock clamps just inside the building. Flexible hose will be used to bridge the connection between the solid 4-inch PVC piping and the existing 3-inch SVE manifold piping in the Treatment Building. The *ex situ* SVE soil treatment cell shall include 2-inch diameter, slotted air inlet pipe(s) and a vapor monitoring point as shown on Drawing C-13.

2.8.2 Treated Water and Wastewater Storage

The existing wastewater storage and transfer system is currently operational and is configured to treat wastewater from either the ECC Site or the neighboring Third Site. During the construction of the augmented SVE system at the ECC Site, the connection to Third Site will be closed and the system will be used solely for ECC water. Thereafter, the wastewater treatment system may be operated by both ECC and Third Site, provided that separate waste streams are maintained. Simultaneous operation by both ECC and Third Site can occur through alternating treatment. As previously noted, an additional raw wastewater tank (T-5) will be required to maintain separate waste streams. Tank T-5 will be constructed using the same design details as existing tank T-2, including liner, cover, diffuser, and leak detection system. The design details for Tank T-5 are presented on Drawing C-14 and in Specification 13050.

Two temporary tanks (T-6 and T-7) will be utilized to collect water from the augmented SVE trenches during initial dewatering. Tank T-6 will be staged near Trench Segment 4 and will receive water from Segments 1, 2, 3, and 4. Tank T-7 will be staged near Trench Segment 6 and will receive water from Segments 5, 6, and 7. Tanks T-6 and T-7 will be equipped with centrifugal transfer pumps. If deemed appropriate, the temporary tanks may be equipped with tank heaters. Electrical service, for both the pumps and the heaters, will be supplied to each water collection tank location. The design details for Tanks T-6 and T-7 are presented in Specification 13050.

A 150,000-gallon clean water tank (T-4) currently exists at the Site. This tank will be used to store treated water prior to discharge Unnamed Ditch. After completion of the Active Phase and the Phase I Monitoring, consideration will be given to removing the treatment system including Tanks 2, 4, and 5 and relying on skid-mounted equipment subject to any need for the treatment facility by Third Site.

2.8.3 Treated Water Discharge Procedures

The Site subsurface water will be treated in batches with sampling of each treated batch conducted prior to discharge. The treated water samples will be analyzed by the Contractor[9] for the parameters listed in the approved effluent limits. Wastewater streams for the ECC Site and Third Site will not be combined. Once treated and in compliance with treated water effluent limits, the wastewater will be discharged to Unnamed Ditch at the Site.

2.9 Monitoring Well Installation

Wells S-4B and S-5 will be installed by the Trust's Engineer after completion of SVE trench construction. Each well will be double-cased through the till as described in Specification 02268 in Appendix A. The well locations are provided in Appendix C.

2.10 Decommissioning/Demobilization

Following completion of the Construction Phase, Site restoration activities and all equipment and materials used to construct the augmented SVE system that are not needed for the Active Phase of operation will be removed from the Site. Site restoration activities to be conducted following construction, prior to the beginning of the Active Phase, include:

[9] Effluent limits were provided by IDEM in 1997 and are included in Table 2-3 and the FSP.

- Removal of all construction and silt fencing.
- Placement of topsoil (as necessary) and seed to restore vegetative cover to all disturbed areas.
- Replacement of fence on eastern boundary of Site, with clearing/grubbing, as needed.
- Clean out and, if needed, repair of drainage channels.
- Removal of any construction trailers and construction equipment not needed for monitoring/operation phases.
- Handling of excavated soil as described in Section 2.8.1.

3.0 ACTIVE PHASE ACTIVITIES

3.1 Augmented SVE System Operations

In accordance with the Attachment Z-1 Remedy, the augmented SVE trench system will operate until the Soil Vapor Standards are achieved. The Contractor shall operate, maintain, and optimize the system for efficient vapor removal and provide regular operations and status reports. Continuous operation of the SVE system, except for temporary shutdowns for maintenance and repairs, is anticipated. Electrical, repair, and soil vapor monitoring costs will be the responsibility of the Contractor. The Contractor shall notify the Trust when Soil Vapor Standards have been met.

The Soil Vapor Standards were presented in Table 2-2.[10] The time required to attain the Soil Vapor Standards is dependent on the adequate removal of water, the initial concentrations of the contaminants of concern (COCs), the minimization of short-circuiting, operating air flow rate and temperature, and the efficient diffusion of air through the soil pores. Based on the previous SVE activities conducted at the Site, the attainment of shutdown standards is expected to occur within 6 months of operation of the extraction systems in the augmented SVE system. However, the actual time may be longer or shorter. If an individual trench segment has reached the Soil Vapor Standards (Table 2-2), the Trust may request approval from the USEPA, in consultation with IDEM, to suspend SVE in that trench segment until the end of the Active Phase in order to better concentrate the augmented SVE system on the remaining trench segments.

3.2 Soil Vapor Monitoring

The augmented SVE system configuration allows collection of vapor samples from each SVE trench segment and from the combined vapor stream from all operating SVE trenches. Vapor samples will be collected in accordance with procedures in the Addendum to the FSP, which is contained in Appendix C. The vapor from each SVE trench will be sampled from the vacuum inlet manifold port daily (except weekends) during the first week of operation, weekly for the following 4 weeks, and biweekly thereafter during the Active Phase. The individual trench vapor samples will be analyzed by an off-site laboratory for the selected volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) listed on Table 2-2 in accordance with the Quality Assurance Project Plan (QAPP) included as Appendix E.

The existing in-line Series 8800 Continuous analyzer will be used to monitor total organics in the combined vapor stream from the active SVE trench segments. A vapor sample from the combined vapor will be sent to an off-site laboratory for VOC and SVOC analyses for the first

[10] Soil Vapor Standards in Table 2-2 are from Table 4-1 of the Revised Exhibit A to the Consent Decree.

5 weekdays of operation. An initial correlation will be developed between the in-line continuous analyzer results and combined vapor samples sent off site for analysis.

The combined vapor flow rate will be monitored and recorded to provide sufficient data to calculate the mass of organics removed from the soils and the effectiveness of the system.

3.3 Surface Water Monitoring

During Active Phase operation of the augmented SVE system, the surface water within the Unnamed Ditch will be monitored semiannually. Surface water samples will be collected upstream and downstream of the Site and at the Northside Landfill discharge location within Unnamed Ditch, as depicted on Figure C-1 in the Addendum to the FSP (Appendix C). Surface water samples will be collected using procedures described in the Addendum to the FSP (Appendix C). The surface water samples will be analyzed for compounds with Acceptable Stream Concentrations (Table 2-1) in accordance with methods and procedures listed in the Addendum to the FSP. If surface water is not encountered, the specific sampling event will be considered complete, despite the inability to gather a full set of data.

3.4 Subsurface Water Monitoring

During operation of the augmented SVE system, the subsurface water within the dewatering wells in the operating augmented SVE trench and monitoring wells S-1, S-4B, and S-5 will be sampled semiannually. The locations of the augmented SVE dewatering wells and wells S-1, S-4B, and S-5 are depicted on Figure C-1 of the Addendum to the FSP (Appendix C). Subsurface water samples will be collected from wells, as described in the Addendum to the FSP (Appendix C).

The subsurface water samples will be analyzed for compounds with Acceptable Stream Concentrations (Table 2-1) in accordance with methods and procedures listed in the Addendum to the FSP. If subsurface water is not encountered in a trench, the specific sampling event for that trench will be considered complete, despite the inability to gather a full set of data.

Water levels will be measured in each of the seven augmented SVE trench dewatering wells; wells S-1, S-4B, and S-5; and the TBCW piezometers semiannually during the Active Phase. The dewatering well data will be used to assess whether the initial dewatering system needs to be reactivated in order to maintain less than 18 inches of water in the bottom of each trench during the Active Phase.

3.5 *Ex Situ* SVE Soil Treatment Cell Monitoring

The soil vapors from the *ex situ* SVE soil treatment cell (if constructed) will be sampled at least monthly during active treatment. The soil vapors extracted from the *ex situ* cell will be sampled for compliance purposes with restart spike testing as described in Section 3.6. When the soil vapors meet the Soil Vapor Standards (Table 2-2), the air flow will be discontinued and the cell

will be covered with topsoil and seeded, as detailed in the Contractor's *Ex Situ* SVE Soil Treatment Plan.

3.6 Active Phase Termination

When the vapor concentrations in all SVE trenches are less than the Soil Vapor Standards, a restart spike will be performed to demonstrate that the Soil Vapor Standards have been achieved. The restart spike procedure will include shutting down the entire trench vapor extraction system for a period of 21 days. Prior to such shutdown, authorization will be obtained from USEPA, in consultation with IDEM. On restarting the vapor extraction system, all SVE trenches will be operated as during normal operations for approximately 30 minutes prior to sampling. The vapor extraction system will then be shut down again so that sampling can be performed under static, non-extraction conditions.

After purging the sampling tubing, a sample of soil vapor will be collected from each of the individual SVE well risers into individual Summa canisters. The sampling period for each canister will be 15 to 30 minutes. These samples will be sent for laboratory analysis.

The analytical results from the seven samples will be compared individually to the Soil Vapor Standards (Table 2-2). If the analytical results from the any of the trenches exceed the Soil Vapor Standards, the SVE of that trench will be reactivated for a period of at least 1 week, before the shut down process described above is repeated.

When the results of laboratory analyses of each of the individual trench well head soil vapor samples collected from two consecutive restart spikes conducted 2 weeks apart show that the soil vapor concentrations meet the Soil Vapor Standards at each trench, a water sample will be collected from the PRGS collection manhole.[11] If the water sample meets the Acceptable Stream Concentrations presented in Table 2-1, then operation of the SVE system will be terminated.

If the water sample collected from the PRGS collection manhole does not meet the Acceptable Stream Concentrations, then operation of the augmented SVE system will continue for an additional 90 days before resampling the trench water. If no water is available in the PRGS collection manhole due to dewatering operations, then operation of the SVE system will be terminated when the restart spike tests meet the Soil Vapor Standards.

Following completion of the Active Phase, Site restoration activities and all equipment and materials used to during the Active Phase that are not needed for the Phase I Monitoring and Phase II Long-Term Monitoring may be removed from the Site. Skid-mounted equipment of adequate size and configuration to meet discharge requirements, along with appropriate power supply, alarm, autodialer, controls and secondary containment will be available if needed after the permanent

[11] This sampling will require opening of the valves between the collection piping (SVE screen) and the PRGS conveyance piping and the valves between the trench segments.

equipment is removed. The skid-mounted system will be able to be operated year-round and will include a wastewater treatment system if needed.

Site restoration activities to be conducted following the Active Phase include:

- Removal of aboveground water pipes and hoses, Tank 5, Tank T-6, and Tank T-7.
- Proper drainage of SVE pipelines.
- Decontamination of all process equipment at the ECC Treatment Building.
- Proper treatment/disposal of any solid residues and decontamination wash water.
- Proper disposal of used carbon and carbon canisters.
- Cleaning of decontamination pad and treatment of decontamination water.
- Removal of any remaining office trailers, toilet facilities, and all Contractor equipment/materials from the Site.

4.0 PHASE I MONITORING

4.1 Phase I Monitoring Overview

The Phase I Monitoring period has been defined as the 1-year period beginning when the Soil Vapor Standards and Acceptable Stream Concentrations have been achieved in the augmented SVE trenches. During this period, the augmented SVE trench system will collect till water that will be conveyed to the PRGS treatment vessel.

4.2 Sampling During Phase I Monitoring

Phase I Monitoring includes quarterly sampling and analysis of surface water (SW-1, NSL-1, and SW-2) and subsurface water (SVE trench segment dewatering wells and monitoring wells S-1, S-4B, and S-5) for 1 year after completion of the Active Phase. Quarterly water level measurements from the TBCW piezometers will also be collected during the Phase I Monitoring period. During Phase I Monitoring, the PRGS will be inspected on a quarterly basis in conjunction with the water sampling. Quarterly reports will be provided to the USEPA and IDEM, which will include analytical results from the analyses performed during the previous quarter and discussion of any Site issues.

The water level within the augmented SVE trench system will be maintained by gravity drainage using the PRGS. Control of the water level within the trench system will control the hydraulic gradient within the till unit across the Site, to prevent the flow of till water around or below the augmented SVE system.

4.3 Phase I Monitoring Termination Criteria

Performance criteria for the Phase I Monitoring have been defined in Attachment Z-1. Attachment Z-1 states the actions to be taken in response to each of the performance criteria are as follows:

- If the quarterly subsurface water samples collected from the augmented SVE trench system contain VOCs at concentrations greater than Acceptable Stream Concentrations (Table 2-1), then the PRGS valves will be closed and the augmented SVE system will be reactivated until the vapor meets the Soil Vapor Standards in Table 2-2. If the SVE system is restarted, the 1-year Phase I Monitoring period will also restart unless otherwise agreed to by USEPA and IDEM.

- If the quarterly water levels collected from the TBCW piezometers show that till water is flowing around the augmented SVE system, then the necessary adjustments will be made to the PRGS, as approved by USEPA, in consultation with IDEM.

- If the quarterly surface water samples collected from Unnamed Ditch immediately downgradient of the Site contain VOCs at concentrations greater than the Acceptable Stream Concentrations (Table 2-1), then the source of these compounds will be investigated, further remediation will be evaluated, and, if determined to be necessary by the Trust or by USEPA, in consultation with IDEM, a proposal will be submitted for approval by USEPA, in consultation with IDEM.

- If the TBCW is found to be leaking, then the wall will be repaired under a plan approved by USEPA, in consultation with IDEM. The quarterly water level measurements to be collected from the TBCW piezometers will be used to confirm the integrity of the TBCW.

- If quarterly sampling of sand and gravel monitoring wells S-1, S-4B, or S-5 show increasing trends in VOC concentrations that are above Acceptable Stream Concentrations (Table 2-1), then the cause of the trends will be evaluated and a report will be submitted for approval by USEPA, in consultation with IDEM, that evaluates the trends and proposes additional remedial actions (if necessary).

During Phase I Monitoring, the Site will be evaluated for surface re-use that is protective of the cap, the subsurface water treatment system (including the PRGS), and otherwise protective of human health and the environment. At the completion of the Phase I Monitoring, the SVE treatment plant and its associated above ground storage tanks and underground piping may be removed (subject to whether they are needed in connection with water treatment at the neighboring Third Site) and Phase II Long-Term Monitoring will begin at the Site.

5.0 PHASE II LONG-TERM MONITORING

5.1 Phase II Long-Term Monitoring Overview

The Phase II Long-Term Monitoring period will follow the completion of the 1-year period of Phase I Monitoring and will be divided into Phase II(a) and Phase II(b). During the Phase II Long-Term Monitoring period, the PRGS will continue to allow a treated outlet for subsurface water that accumulates within the augmented SVE trench segments. Monitoring will be conducted as described below.

5.2 Phase II(a) Long-Term Monitoring Criteria

5.2.1 Phase II(a) Long-Term Monitoring

Phase II(a) Long-Term Monitoring will last for 2 years and follow the same sampling schedule as used in Phase I Monitoring.[12]

5.2.2 Phase II(b) Long-Term Monitoring

After successful completion of the 2-year Phase II(a) Monitoring period, the remainder of the Phase II Long-Term Monitoring (Phase II(b)) will commence. The Phase II(b) Monitoring includes maintenance of the PRGS system and monitoring of the PRGS effluent on an annual basis. If inspection of the PRGS indicates bypass, blockage, or other conditions that could restrict or inhibit its performance, the PRGS will be repaired and/or reconditioned, in accordance with a plan approved by USEPA, in consultation with IDEM. If the PRGS effluent exceeds the effluent limits for discharge to Unnamed Ditch in Table 2-3, the PRGS "bed" will be replaced.

If the fresh PRGS bed cannot meet the required standards, additional measures to meet the standards will be evaluated and PRGS monitoring frequency may be temporarily increased to quarterly. Phase II(b) Long-Term Monitoring will continue on a quarterly schedule until USEPA agrees, in consultation with IDEM, that annual sampling may be resumed or the USEPA approves cessation of monitoring.

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[12] If an exceedance of the Acceptable Stream Concentrations is present in the trench dewatering wells during Phase II(a), the valves to the PRGS will be closed and the augmented SVE system will be reactivated or an appropriate skid-mounted system will be activated. When the soil vapor meets the Soil Vapor Standards, the PRGS valves will be opened and the Phase II(a) monitoring period will restart.

6.0 SCHEDULE

Upon USEPA approval of this Design Report, invitations to bid will be sent to potential Contractors. After receipt and evaluation of bids, the Trust will enter into a Contract with the selected Contractor. The Trust will advise USEPA, in writing, of its proposed Contractor after receipt and evaluation of bids (but prior to formally signing a Contract) to ensure that USEPA has no objections to the proposed Contractor.

The Contractor will be required to prepare the final Design Submittals, the project Health and Safety Plan, work schedule, and other required plans required in the Technical Specification (Appendix A) (collectively the "Contractor Submittals") after the contract is awarded. After review by the Trust, the Contractor Submittals will be submitted to the USEPA. The augmented SVE trench system field tasks will be implemented in general accordance with the approved work schedule.

TABLES

TABLE 2-1

Site-Specific Acceptable Concentrations
Enviro-Chem Superfund Site
Zionsville, Indiana

Parameter	Acceptable Subsurface Water Concentration [1] (ug/L)	Acceptable Stream Concentration [2,3] (ug/L)	Acceptable Soil Concentration [4,5] (ug/kg)
Volatile Organic Compounds			
Acetone	3,500 RB		370,000
1,1-Dichloroethene	7 MCL		42,000
1,2-Dichloroethene (total)	70 MCL	7.4 SB	5,800
Ethylbenzene	680 MCL	3,280	160,000
Methylene chloride	133 SSB	15.7	1,800
Methyl ethyl ketone	170 LDWHA		250,000
Methyl isobutyl ketone	1,750 RB		75,000
Tetrachloroethene	0.69 RB	8.85	640
Toluene	2,000 MCL	3,400	240,000
1,1,1-Trichloroethane	200 MCL	5,280	280,000
1,1,2-Trichloroethane	0.61 RB	41.8	300
Trichloroethene	5 MCL	80.7	82
Vinyl chloride	2 MCL	525	13
Xylenes (total)	10,000 MCL		170,000
Semivolatile Organic Compounds			
Bis (2-ethylhexyl) phthalate	7.3 SSB	50,000	
Di-n-butyl phthalate	3,500 RB	154,000	
1,2-Dichlorobenzene	600 MCL	763	220,000
Diethyl phthalate	28,000 RB	52,100	
Isophorone	8.5 RB		
Naphthalene	14,000 RB	620	
Phenol	1,400 RB	570	160,000
Inorganic Parameters			
Antimony	15.6 SSB		
Arsenic	50 MCL	9.2 SB	
Barium	1,000 MCL		
Beryllium	4 MCL		
Cadmium	10 MCL		
Chromium VI	50 MCL	77.6 SB	
Lead	50 MCL	19.8 SB	
Manganese	7,000 RB		
Nickel	150 LDWHA	100	
Silver	50 MCL		
Tin	21,000 RB		
Vanadium	245 RB		
Zinc	7,000 RB	123 SB	
Cyanide (total)	7,000 RB	17.2 SB	
Polychlorinated Biphenyls			
Aroclor 1016	0.6 SSB	0.5 SB	
Aroclor 1221	1.1 SSB	0.9 SB	
Aroclor 1232	0.6 SSB	0.5 SB	
Aroclor 1242	0.6 SSB	0.5 SB	
Aroclor 1248	0.6 SSB	0.5 SB	
Aroclor 1254	0.6 SSB	0.5 SB	
Aroclor 1260	0.6 SSB	0.5 SB	

TABLE 2-1

Site-Specific Acceptable Concentrations
Enviro-Chem Superfund Site
Zionsville, Indiana

Notes:

[1] RB = Risk Based standard. USEPA, Risk Assessment Guidance for Superfund: Volume I - Human Health Evaluation Manual (Part B, Development of Risk-based Preliminary Remediation Goals), December 1991.

MCL = Drinking Water Maximum Contaminant Level (40 CFR 141).

SSB = Applicable Subsurface Water Background Concentrations as defined as two standard deviations above the calculated mean of 12 sample sets of background subsurface water samples. Background subsurface water samples were collected from wells T-5 and S-1 (see Attachment Z-1, Appendix E, Tables E-1 and E-3 for calculations).

LDWHA = Lifetime Drinking Water Health Advisory. USEPA, Superfund Public Health Evaluation Manual update of November 16, 1987.

[2] SB = Applicable Surface Water Background Concentrations were defined as two standard deviations above the calculated mean of 12 sample sets of background surface water samples. Background surface water samples were collected from the surface water sample location SW-1 (see Attachment Z-1, Appendix E, Tables E-2 and E-4 for calculations).

[3] Stream Criteria, from Table 1 of the Record of Decision for the site, September 25, 1987 (or calculated on the same basis) unless otherwise noted.

[4] Acceptable Soil Concentrations are the minimum IDEM RISC non-default closure levels for commercial/industrial soil direct contact, commercial/industrial soil migration to groundwater, construction soils, and the soil saturation limit (see Table 2-1 for calculations).

[5] The Acceptable Soil Concentrations, within the meaning of Exhibit A and the Consent Decree, will be achieved when the arithmetic average of the soil sample results for each parameter, assigning all nondetect results a value of 1/2 the detection limit, do not exceed the values set forth in this table by more than 25%.

TABLE 2-2

Soil Vapor Standards
Enviro-Chem Superfund Site
Zionsville, Indiana

Compound	Soil Vapor Standard	
	(mg/L)	(ppmv)
Volatile Organic Compounds (VOCs):		
Acetone	0.61	244
1,1-Dichloroethene	2	481
1,2-Dichloroethene (total)	3.7	880
Ethylbenzene	37	8,076
Methylene chloride	0.08	22
Methyl ethyl ketone	0.04	13
Methyl isobutyl ketone	0.69	159
Tetrachloroethene	0.11	16
Toluene	107	27,090
1,1,1-Trichloroethane	8.3	1,442
1,1,2-Trichloroethane	0.01	1
Trichloroethene	0.39	68
Vinyl chloride	919.2	338,808
Total Xylenes	595	130,244
Base Neutral/Acid Organics:		
1,2-Dichlorobenzene	9.3	1,466
Phenol	0.005	1.3

Source: Table D-1 of Revised Exhibit A, Revision 2, May 7, 1997.

TABLE 2-3

Effluent Limits for Discharge of Treated Water to Unnamed Ditch[1]
Enviro-Chem Superfund Site
Zionsville, Indiana

Contaminant of Concern (COC)	Discharge Limit (ug/l)
1,1-Dichloroethane	990 [2]
1,1-Dichloroethene	2
Cis -1, 2-Dichloroethene	2
Trans-1, 2-Dichloroethene	2
Tetrachloroethene	5
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	42
Trichloroethene	10
Vinyl Chloride	10
bis(2-Ethylhexyl)phthalate	0.68
Di-n-butylphthalate	0.021
Diethylphthalate	7
1, 2-Dichlorobenzene	0.76
Naphthalene	0.069
Phenol	0.57

ug/l = micrograms per liter

[1] Effluent limits from February 1997 *Briefing Memo: Environmental Conservation and Chemical Zionsville, Indiana Superfund Site, ARAR Effluent Limits*, prepared by George Oliver, IDEM Office of Water Management.

[2] Effluent limits not set forth in IDEM Briefing Memo. Value represents IDEM Tier 1 Default Ground Water Residential Criterion.

TABLE 2-4

Summary of ECC Augmented SVE System Alarms
Enviro-Chem Superfund Site
Zionsville, Indiana

System Component	Alarm Condition	Alarm Instrumentation	Alarm Type / Notification
Dewatering Wells	High water in trench	Transducer in SVE trench segment dewatering wells connected to CU300 controller	page operator
	Failure of pump or transducer	PLC	page operator
Temporary Water Storage Tanks T-6 and T-7	High water in Tank T-6 or T-7	Float switch and controller box	shut down SVE dewatering pumps, page operator
	Failure of pump, heater or switch controller	PLC	shut down dewatering pumps, page operator
New Tank T-5	High water in Tank T-5	Transducer	shut down pumps, page operator
	High water in leak detection sump	Transducer	page operator
PRGS Collection Manhole	High water in manhole	Transducer and CU300 (use from dewatering well)	page operator
	Failure of pump or tranducer	PLC	page operator
SVE Blower System	SVE entrainment separator (knockout tank) High Level	float switch at knockout tank	shut down blower, page operator
	High organics	in-line organic vapor analyzer at carbon vessel	shut down blower, page operator to change GAC
Treatment Building	Power failure	PLC	page operator
	Floor Sump	float switch in sump	shut down pumps, page operator

TABLE Z-5

Site-Specific Soil Exposure Concentrations[1]
Enviro-Chem Superfund Site
Zionsville, Indiana

Parameter	Soil Saturation Limit C_{sat} (mg/kg)	Commercial/Industrial Soil Direct Contact		Commercial/Industrial Soil Migration to Groundwater		Construction Soils	
		Carcinogen C_{ssic} (mg/kg)	Non-carcinogen C_{ssin} (mg/kg)	Carcinogen C_{sbsic} (mg/kg)	Non-carcinogen C_{sbsin} (mg/kg)	Carcinogen C_{sscc} (mg/kg)	Non-carcinogen C_{sscn} (mg/kg)
Acetone	201,292	NE	13,987	NE	370	NE	72,936
1,1-Dichloroethene	930	NE	738	NE	42	NE	3,861
cis-1,2-Dichloroethene	1,001	NE	246	NE	5.8	NE	1,240
trans-1,2-Dichloroethene	2,138	NE	392	NE	14	NE	2,012
Ethylbenzene	160	NE	9,129	NE	195	NE	36,644
Methylene chloride	3,008	355	12,472	1.8	28	35,203	36,962
Methyl ethyl ketone	28,194	NE	119,270	NE	250	NE	360,827
Methyl isobutyl ketone	8,750	NE	31,537	NE	75	NE	65,900
Tetrachloroethene	115	40	214	0.64	12	4,258	1,093
Toluene	309	NE	3,174	NE	240	NE	16,531
1,1,1-Trichloroethane	642	NE	10,663	NE	280	NE	51,059
1,1,2-Trichloroethane	1,342	24	194	0.30	2.5	2,786	896
Trichloroethene	627	1.7	93	0.082	0.35	211	224
Vinyl chloride	928	4.8	262	0.013	2.1	319	1,064
Xylenes (total)	170	NE	1,242	NE	426	NE	6,724
1,2-Dichlorobenzene	220	NE	5,103	NE	265	NE	22,889
Phenol	21,329	NE	113,262	NE	160	NE	243,091

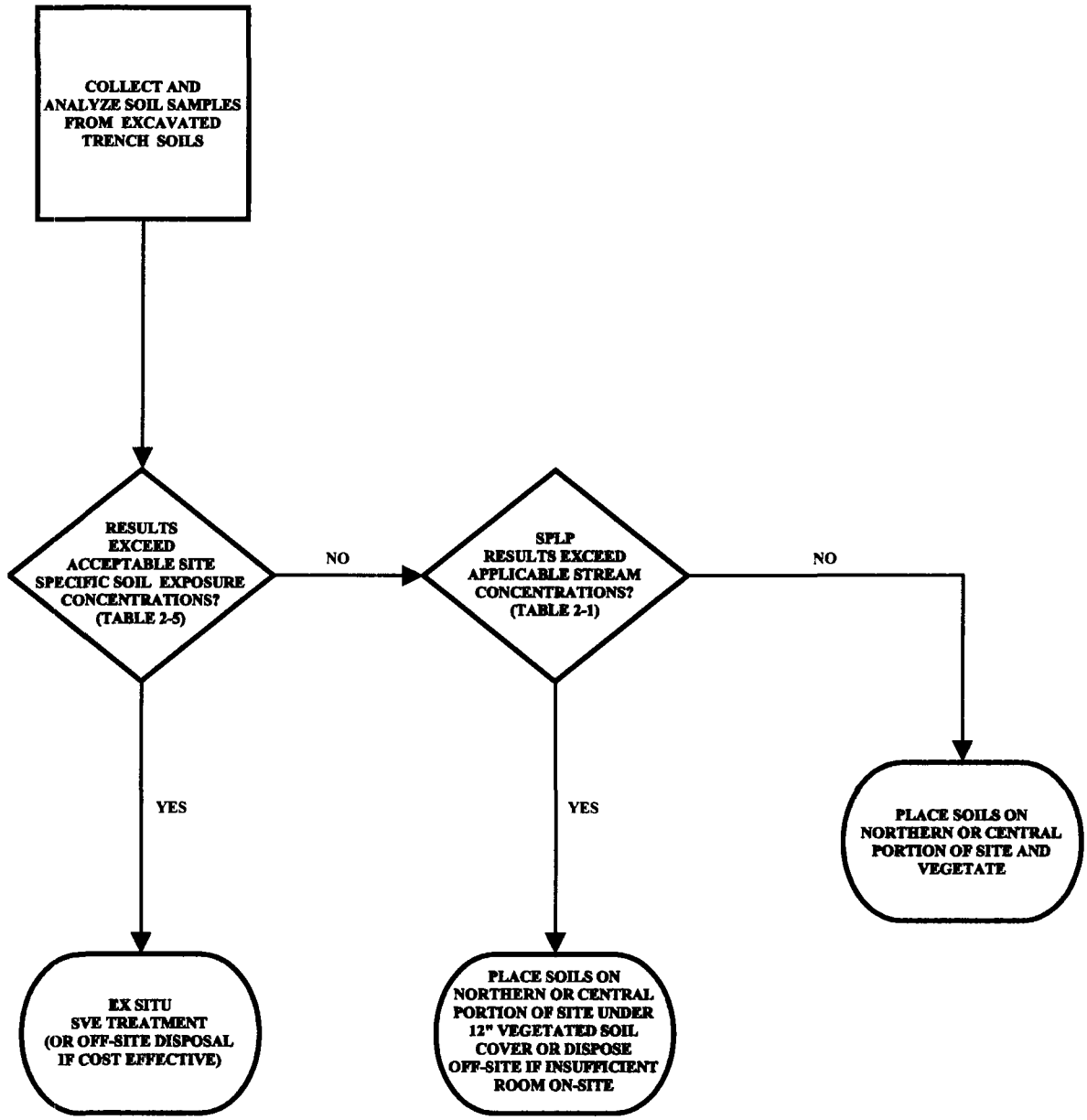
Legend:

C_{sat} Soil Saturation Limit (Attachment Z-1, Appendix D, Table C, Equation 7-3)
C_{ssic} Commercial/Industrial Soil Closure Level for Direct Contact for Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-9)
C_{ssin} Commercial/Industrial Soil Closure Level for Direct Contact for Non-Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-10)
C_{sbsic} Commercial/Industrial Migration to Ground Water Contact Closure Level for Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-11)
C_{sbsin} Commercial/Industrial Migration to Ground Water Contact Closure Level for Non-Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-12)
C_{sscc} Construction Soil Closure Level for Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-13)
C_{sscn} Construction Soil Closure Level for Non-Carcinogens (Attachment Z-1, Appendix D, Table C, Equation A1-14)
NE Not established

Notes:

[1] Default IDEM RISC equation parameters/exposure assumptions were used for the closure level calculations as included in Attachment Z-1, Appendix D, Table D. All default exposure equations and human health toxicity default values were used for the calculations as included in Attachment Z-1, Appendix D, Tables C and F.

FIGURE



R:\Client Project Files\EC_21-6585\Additional Work per Exhibit A\ASVES Design_Jan2007\Figures\2-1 Excavated Soils Management Decision Tree.dwg

ENVIRON

EXCAVATED SOILS MANAGEMENT DECISION TREE
ENVIRO-CHEM SUPERFUND SITE
985 U.S. HIGHWAY 421
ZIONSVILLE, INDIANA

Figure
2-1

Drafter: APR | Date: 02/02/07 | Contract Number: 21-6585K | Approved: | Revised:

APPENDIX A

Technical Specifications

TECHNICAL SPECIFICATIONS

CONTENTS

SECTION 01010

SUMMARY OF WORK

SECTION 01010

SUMMARY OF WORK

PART 1 – GENERAL

1.01 SCOPE

A. This section includes a brief description of the major components covered under this contract. The scope of work includes both construction and operation of the remedial action. A more complete description of the work is provided in individual sections of these Specifications and on the Drawings. The Contractor shall furnish all equipment, labor, materials, health and safety, quality control services, and execution of all work necessary to complete the work for final acceptance as outlined in the Design Report.

B. Background. This removal action is subject to the requirements of the Consent Decree for "Additional Work" and Attachment Z-1 between the United States Environmental Protection Agency (USEPA) and the ECC Potentially Responsible Parties (PRPs).

1.02 GENERAL REQUIREMENTS

A. As minimum requirements, the Contractor shall observe and comply with all applicable federal, state, and local laws, rules and regulations in conducting the work. The Contractor shall be responsible for contacting and informing the proper federal, state, and local agencies of the nature and timing of work activities and for securing all necessary and applicable permits required to perform the work covered by this contract.

B. The Contractor shall protect utility lines and/or appurtenances as well as the cap over the Northern and Central SVE (soil vapor extraction) Areas. It is the Contractor's responsibility to locate existing utilities on site. Any damage shall be repaired by the Contractor at no expense to the Environmental Conservation and Chemical Trust Fund (the "ECC Trust"). Any required removal and replacement for construction access or other reasons shall also be the responsibility of the Contractor.

C. Materials and equipment shall be adequate in capacity for the required usage, must not create unsafe conditions, and shall meet the requirements of all applicable codes and standards.

1.03 DESCRIPTION OF WORK

A. The steps required of the Contractor are described in the Design Report, final Design Submittals, the Design Drawings and the Contract. The steps include the implementation of the tasks outlined in Attachment Z-1 to achieve the cleanup levels described therein and includes the following work.

1.04 SUBMITTALS

As specified in project specifications and Design Report.

1.05 PROJECT/WORK IDENTIFICATION

1. Site preparation activities, which include all necessary clearing and grubbing activities, installing temporary construction access driveway, providing electrical hookups and electricity, where necessary, and installing/repairing erosion and sediment controls.

2. Temporary Site Facilities: Provide and maintain temporary site facilities during the performance of this contract such as office trailers, sanitary facilities, security, and the personnel decontamination area.

3. Site Plans: The Contractor shall prepare and implement a Contractor Health and Safety Plan, Contractor Quality Control Plan, and a Site Operations and Maintenance Addendum to the existing Operations and Maintenance Plan for use during construction and operation of the augmented SVE system. The Contractor shall also be responsible for adherence to the Addendum to the Field Sampling Plan (where applicable), other approved plans (Decontamination Plan, Spill Control Plan, Erosion Control Plan, Off-site Disposal Plan, Augmented SVE Trench Construction Plan), and the approved Project Schedule. An *Ex Situ* SVE Soil Treatment Configuration Plan shall be submitted for approval as soon as possible after determination of the soil volume (if any) to be treated.

4. Construct on-site wastewater storage tank and fit to the existing wastewater treatment system (with new carbon vessels), and a temporary water transfer system.

5. Upgrade existing ECC SVE vacuum system as required for operation of the facility for the duration of the program.

6. Install new SVE trench sections to augment existing system.

7. Construct *ex situ* soil treatment cell, if needed.

8. Install a permeable reactive gate (with water carrier pipe system) to be used at completion of the SVE operations.

9. Operate and maintain the SVE system, including initial dewatering of the SVE trenches, until the Soil Vapor Standards set forth in Table 3-1 of the Design Report are achieved; including treatment/disposal of wastewater and electricity costs.

10. Submittal of an "as-built" report (Contractor Submittal) upon completion of Active Phase construction activities. All required Contractor-prepared post-completion documentation set forth in the Technical Specifications shall be submitted according to the agreed-upon schedule. In addition to the information required in the Technical Specifications and the Construction Submittals, the Contractor shall provide quantities and types of materials removed from off-site or handled on-site, methods of removal and disposal, the ultimate destinations of disposed materials, and other relevant documentation generated during the removal action.

11. Demobilization of Site of all Contractor equipment, and removal of the temporary facilities after completion of the Active Phase and site restoration. The Contractor shall leave the site security fence and gates, equipment decontamination pad, the wastewater storage pad, and utilities on-site after shutdown of SVE System operations. At the completion of the construction work, all temporary site facilities shall be removed from the Site, except those that are required during the operations phase or as requested by the Trust.

12. All other activities to satisfactorily complete all work covered by these Specifications, final Design Drawings, and the final Design Report not specifically discussed but necessary for the project construction and final acceptance. All other work required by the ECC Trust under the terms of this contract.

1.07 AVAILABLE SITE INFORMATION

A. The following background documents shall be provided by CD ROM disk. The additional documents listed below provide background information and supporting technical rationale upon which the specifications and drawings are based and do not contain additional work requirements.

1. November 1997 100% SVE and WWT Design and drawings
2. CPT logs/elevations
3. Attachment Z-1
4. Consent Decree
5. Revised Remedial Action (Exhibit A), Revision 2, May 7, 1997

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 01015

SEQUENCE OF WORK

SECTION 01015

SEQUENCE OF WORK

PART 1 – GENERAL

1.01 GENERAL SEQUENCE OF WORK ACTIVITIES

A. The individual work tasks at the Site shall be conducted in the general sequence indicated in this section. The general sequence includes both concurrent operations and operations that must be completed before or after other construction activities. Except as provided in Part B below, the sequence of work shall not be changed without the prior written approval of the ECC Trust's Engineer (the "Engineer").

The Contractor is hereby notified that the site support zone, material storage areas and work area between the existing cap and the east slope are very limited in areal extent as shown on the drawings. Materials and equipment logistical planning and scheduling are critical to successful implementation of the work.

B. Changes to the general sequence of work shall be approved by the Trust prior to the Contractor's submittal of his final work schedule.

C. The following general sequence of work shall be used on the project (see other Technical Specification and Design Report for details of work):

1. Preparation of project submittals. The Contractor will assist the Trustees in negotiating to obtain IDEM and USEPA approval of the submittals.

2. Site preparation

a. Mobilization of Contractor equipment, personnel, and temporary facilities required for construction activities to the Site after the ECC Trust gives the Contractor written notice to proceed with construction:

b. Installation of erosion controls and temporary access roads.

c Utility relocation, as needed.

d. Installation of new water storage tank, T-5, prior to need for water storage.

3. Construction

   a. Install SVE Trenches with SVE and PRGS piping. Note that a buried sheet pile wall exists in the vicinity of Trench Segments 4 and 5, which may need to be removed or cut off during ASVES trench construction.

   b. Install dewatering equipment, wells, pumps, temporary tanks, water lines.

   c. Construct *ex situ* soil treatment cell, if needed, install SVE piping to vacuum system, construct PRGS vault/infiltration area.

   d. Repair any damage to existing cap or cap, reseed, regrade, as needed.

4. Testing

   a. Perform dewatering test prior to acceptance of SVE trench construction.

5. Operations

   a. Initial dewatering of SVE Trenches.

   b. SVE operations with dewatering as needed; concurrent *ex situ* treatment cell operation, if needed.

   c. Upon completion of SVE activities, connect SVE screen to PRGS conveyance piping and open connection to PRGS treatment vault.

6. Demobilization (at end of Active Phase)

   a. Removal of temporary facilities; replacement and restoration of site fencing, vegetative cover, etc.

   b. Removal of Contractor equipment and personnel from the Site.

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 01050

GRADES, LINES, AND LEVELS

SECTION 01050

GRADES, LINES, AND LEVELS

PART 1 – GENERAL

1.01 DESCRIPTION

A. All work under this Contract shall be performed in accordance with the lines and grades shown on the Design Drawings or as directed by the Trust's Engineer. Plans of existing site conditions are included in the Design Drawings. It shall be the responsibility of the Contractor to verify that the plans furnished are a true representation of the conditions of the site. Any error or apparent discrepancy in the data shown, or omissions of data required for accurately accomplishing the stakeout survey shall be referred immediately to the Trust's Engineer for interpretation or correction.

B. All survey work shall be performed by the Contractor at its expense. The Contractor shall employ a land surveyor registered in the State of Indiana that has competently qualified personnel and all necessary instruments, stakes and other material to perform the work. While its use is not mandatory, the following engineering firm is familiar with the site and the site benchmarks:

Schneider Engineering
Historic Fort Harrison
8901 Otis Avenue
Indianapolis, IN 46216

C. Contractor shall establish all base lines for the location of the principal component parts of the Work, together with a suitable number of benchmarks. Based upon the information provided by the Design Drawings, the Contractor shall develop and make all detail survey stakes for all working points, lines and elevations. Locations of the augmented SVE trenches, and permeable reactive gate system shall be laid out and later confirmed by a licensed surveyor.

D. The Trust's Engineer may check all or any portion of the Work, and the Contractor shall afford all necessary assistance to the Trust's Engineer or their subcontractor in carrying out such checks. Any necessary corrections to the Work shall be immediately made by the Contractor at no additional cost to the Trust. Such checking by the Trust's Engineer shall not relieve the Contractor of any responsibilities for the accuracy or completeness of its work.

E. Contractor shall be responsible for informing the surveyor regarding the Health and Safety requirements and enforcing such requirements.

1.02 SUBMITTALS

A. Name, address, telephone number of the surveyor to the Trust's Engineer at least 1 week prior to the start of survey activities.

B. Valid Insurance Certificate for the surveyor at least 1 week prior to the start of survey activities.

1.03 QUANTITY SURVEYS

A. No quantity surveys are anticipated.

104. QUALITY CONTROL

A. The Contractor shall employ a land surveyor registered in the State of Indiana and acceptable to the Trust's Engineer Representative to perform the survey work.

B. Contractor shall submit a valid insurance certificate for the surveyor.

C. All coordinates and elevations shall be surveyed to 0.01 feet.

D. All survey coordinates shall be correlated to the site benchmark.

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 01210

PRE-CONSTRUCTION WORK CONFERENCE

SECTION 01210

PRE-CONSTRUCTION WORK CONFERENCE

PART 1 – GENERAL

1.01 SCOPE

A. This section covers the conference required after the Notice to Proceed but prior to commencing with construction.

1.02 PRE-CONSTRUCTION WORK CONFERENCE

A. Prior to any site work being formed, a Pre-Construction Work Conference will be held between the Contractor, Trustees, the Trust's Engineer and any major subcontractors. Attendance by the Contractor's Site Superintendent, quality control personnel, and safety personnel will be required. The USEPA and IDEM will be invited to attend.

B. The purposes of this conference are to further define the quality control system, review Contractor submittals and discuss project sequencing. The specifics of the Contractor's emergency procedures and health and safety requirements will be presented so that they are understood by all those directly related to the site work. Other Contractor procedures will also be discussed and any modifications will be discussed.

C. At least 10 working days prior to the Pre-Construction Work Conference, the Contractor shall submit to the Trust's Engineer the required project submittals. The CQC Plan will be reviewed to provide an understanding of the quality control system. The Contractor's Progress Schedule will be discussed. Questions concerning administrative requirements, chain of command or any other aspect of the project may also be addressed.

D. Pre-Construction submittals shall include (but not be limited to):

1. Contractor Site-Specific Health and Safety Plan
2. Spill and Contingency Control Plan
3. Erosion and Sediment Control Plan
4. PRGS materials supplier
5. Augmented SVE Trench Construction Plan, including slurry specialist resume
6. Manhole specifications

In addition, if off-site disposal is required, an Off-site Disposal Plan shall be required prior to removal of wastes.

E. The Pre-Construction Work Conference shall be arranged by and paid for by the Contractor at a location and date acceptable to the ECC Trust representatives.

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 01390

HEALTH AND SAFETY PLAN

SECTION 01390

HEALTH AND SAFETY PLAN

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Contractor shall develop a Site-Specific Health and Safety Plan (HASP) for the Work which at a minimum complies with the project Health and Safety Plan.

1.02 RELATED WORK NOT INCLUDED

The Site-Specific HASP shall include all phases of the Work.

1.03 SUBMITTALS

A. Site-Specific HASP shall be submitted to the Trust at least 10 days prior to the Pre-Construction Conference.

1.04 PERFORMANCE OF WORK

A. All workers at the site must have current OSHA HAZWOPER 40-hour training for all intrusive activities (e.g., excavation, soil/ground water/waste handling, etc.) and any other activities during which workers may be exposed to potentially contaminated materials (e.g., equipment decontamination).

B. Contractor shall have exclusive and complete control over his employees, subcontractors and vendors and shall be solely responsible for their acts. Trust's Engineer may notify Contractor when any employee on the project site is, in his opinion, incompetent or disorderly, or has refused to carry out the provisions of the Contract, or has used threatening or abusive language to any person on the work, or is otherwise unsatisfactory. Upon Contractor's receipt of such notice, such employee shall be discharged immediately from the work and shall not be employed again on the project, except with the written permission of the Trust's Engineer.

C. Contractor is solely responsible for compliance with health and safety requirements associated with the Work. The Trust and the Trust's Engineer will not be held responsible in any way for the Contractor's compliance with all or any standards and regulations promulgated under the Federal Occupational Safety and Health Act of 1970 and other similar state or local statutes.

1.05 SITE SPECIFIC HEALTH AND SAFETY PLAN (HASP)

A. The Contractor shall be responsible for the development and implementation of a site-specific HASP in order to ensure safe operations for all aspects of work covered by this Contract. The plan shall be based on all applicable Occupational Safety and Health Administration (OSHA) standards and regulations. At a minimum, the Contractor's HASP shall identify a safety and health officer and shall address the following items:

1. Identification of work zones for personnel protection;
2. Training of Contractor personnel;
3. Medical surveillance;
4. Communication;
5. Emergency and first aid requirements;
6. Personal safety clothing and equipment;
7. Personal hygiene;
8. Air monitoring (personnel) and action levels;
9. Regulated material control;
10. Dust control;
11. Personnel cleanup procedures;
12. Handling and transportation of materials;
13. Trenching and shoring procedures;
14. Confined space entry (if applicable);
15. Contingency plans for adjustment of work procedures for protection of Trust's Engineer, Trust's representatives, workers, adjacent residence occupants, and transients; and
16. Emergency response plan.

B. The Site-Specific HASP shall comply with OSHA 29 CFR 1904, 1910, 1926, and 1929.

C. Contractor shall submit the HASP to the Trust's Engineer at least 10 days prior to the Pre-Construction Work Conference. The Contractor shall revise and re-submit, if necessary, the HASP addressing the Trust Engineer's comments.

D. The HASP shall comply with the Project Health and Safety Plan prepared by the Trust's Engineer which will be made available to the Contractor for review.

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 01460

SPILL CONTROL

SECTION 01460

SPILL CONTROL

PART 1 – GENERAL

1.01 DESCRIPTION

A. Contractor shall provide equipment to perform emergency measures required to contain any spillage and to remove spilled materials and soils or liquids that become contaminated due to spillage. This collected spill material will be properly handled at the Contractor's expense. This includes spillage during excavation, construction of components described in the Design Report, loading/unloading equipment, on-site fueling of equipment, decontamination, and pumping/transport of subsurface water and other waste materials.

1.02 RELATED WORK NOT INCLUDED

A. Section 01390 – Health and Safety Plan

1.03 SUBMITTALS

A. Spill Control and Contingency Plan; submit to Trust's Engineer for review and comment at least 10 days prior to the Pre-Construction Conference.

B. Spill Response Report, if necessary.

PART 2 – PRODUCTS

2.01 MATERIALS

A. Contractor shall provide means, methods, and facilities required to prevent contamination of Unnamed Ditch, Site drainage ditches, Finley Creek on-site and off-site soil and subsurface water, uncontaminated structures, equipment or material by spills from Site operations.

B. Contractor shall provide control of any unexpected spills by providing the following minimum equipment on site at all times during work activities:

1. Clean fill, or other non-combustible absorbent;
2. Shovels; and
3. Wash water for decontaminating tools and equipment.

PART 3 – EXECUTION

3.01 SPILL CONTROL AND CONTINGENCY PLAN

A. Contractor shall take measures to control and contain any spills immediately, including but not limited to the following:

1. Isolate hazardous areas and keep unnecessary personnel away and, if necessary, up wind.

2. Prevent personnel from contacting spilled material without proper personal protective equipment.

3. Use foam suppressant to reduce vapors and dust, as needed.

4. Use containment and diversionary measures as required to prevent spills from entering surface waters, ditches and drains.

5. Identify all on-site above ground tanks and take measures to protect against and control any spills.

B. Contractor shall provide curbing or loading pad as needed to protect the Site diversion ditches, Unnamed Ditch, Finley Creek, support zones and access roads from spillage from supply trucks or tanks, during mixing/installation of biopolymer slurry, during water transfer, equipment fueling or other construction activities.

C. Decontamination Procedures:

1. Decontamination may be required after cleanup to eliminate traces of the substance spilled or reduce it to an acceptable level as determined by the Trust's Engineer.

2. Contractor shall provide equipment and personnel to perform decontamination measures that may be required to remove spillage from previously uncontaminated structures, equipment, material or existing ground. Cleanup may require removal of previously uncontaminated soil.

3. Personnel decontamination may include showers and cleaning or disposing of clothing and protective equipment, as appropriate.

D. Spill Reporting:

1. After each spill, the Contractor shall assess whether a "reportable spill" has occurred based upon the material spilled and quantity. If needed, a spill report that identifies the date, location, and cause of each spill, the type and quantity

of material spilled, and the corrective action taken, should be submitted to the Trust's Engineer appropriate regulatory authorities.

2. For a reportable spill, the spill report must be submitted within 24 hours after the spill occurred. If a reportable spill occurs, the Trust's Engineer must be notified of the spill before the end of the day of the spill.

END OF SECTION

SECTION 01502

ENVIRONMENTAL PROTECTION

SECTION 01502

ENVIRONMENTAL PROTECTION

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Contractor shall furnish all labor, equipment, and materials required for environmental protection during and as the result of construction operations under this Contract, Design Report and Design Drawings. Environmental protection requires consideration of air, water and land, and involves noise and solid waste management as well as other pollutants. Contractor's personnel performing intrusive work must have current OSHA 40-hour training for hazardous waste operations.

1.02 SUBMITTALS

A. The Contractor shall prepare and submit an Erosion and Sediment Control Plan to the Trust's Engineer. The plan shall follow the requirements herein specified.

1.03 NATURAL ENVIRONMENT

A. The Contractor shall comply with all state and federal laws, rules and regulations pertaining to the preservation and protection of the natural environment, and bear fully all civil and/or criminal penalties that may arise from the work operations of Contractor, whether such penalties are assessed against the Trust, Trust's Engineer, or Contractor.

B. The Contractor shall conduct its activities and shall program its intrusive and restorative operations in such a manner as to minimize pollution of Finley Creek, Unnamed Ditch, and adjacent driveways from erosion of excavated or exposed soils, and backfilled material during periods of excavation and during periods of surface water runoff. The Contractor shall reduce the area and duration of exposure of all erodible soils to the maximum extent practicable. If used, pipeline berms shall be protected by erosion control blankets or equivalent. If damaged, diversion channels shall be lined with concrete revetment, riprap or equivalent.

1.04 SOIL EROSION AND SEDIMENT CONTROL PLAN

The plan shall include location and construction details of the Contractor's measures proposed to control erosion and sedimentation and include measures to maintain those controls throughout the project until site restoration is complete (i.e., when final seeding is established).

PART 2 – PRODUCTS

2.01 GENERAL

A. All materials shall be in accordance with the plans for environmental protection and shall conform to federal, state, and local regulations.

2.02 MATERIALS

A. The following materials may be used in conjunction with this Work, for specific erosion and sediment control applications:

1. Silt Fence
2. Hay Bales
3. Topsoil
4. Riprap
5. Concrete Structures
6. Absorbent Socks
7. Crushed Stone
8. Silt Fence Geotextile

PART 3 – EXECUTION

3.01 GENERAL

A. It is the Contractor's responsibility to determine the means and methods necessary to fully comply with the requirements of this section.

B. It is intended that the resources within the project boundaries and outside the limits of permanent work performed as described in the Design Report be preserved in their present condition, or be restored to a condition after completion of construction, that will appear to be natural and not detract from the appearance of the project. The contractor shall confine its construction activities to areas defined on the Design Drawings except with written approval of the Trust's Engineer.

C. Limits of working areas include areas for storage of construction material, and shall be cleared in a manner which will enable satisfactory restoration and which will not affect the environment during or after the construction period. The Contractor shall not enter beyond the working limits of the working area except with written approval of the Trust's Engineer and/or the private property owner(s).

D. The location of areas for storage of the Contractor's materials required temporarily in the performance of the Work, shall be within the limits of the Site and Support Zone, or temporary storage areas established for this Work.

3.02 PROTECTION OF WATER RESOURCES

A. The Contractor shall not pollute streams, lakes or reservoirs with bentonite, bentonite-type products such as Impermix, guar slurry, fuels, oils, bitumens, hydrogen peroxide, calcium chloride, acids or any other harmful materials used or generated during completion of the removal action. It is the responsibility of the Contractor to investigate and comply with all applicable Federal, State, County and Municipal laws and regulations concerning pollution of rivers, streams and impounded water. All work under this Contract shall be performed in such a manner that objectionable conditions will not be created in streams through, or bodies of water adjacent to, the project area.

B. Surface drainage from cuts and fills within the construction limits, whether or not completed shall be graded to control erosion within acceptable limits. Temporary erosion and sediment control measures such as berms or dikes, if required to meet the above standards, shall be provided and maintained until permanent drainage and erosion control facilities are completed and operative. The area of bare soil exposed at any one time by construction operations should be held to a minimum.

C. At all times of the year, special measures shall be taken to prevent chemicals, fuels, oils, greases, bituminous materials, waste washing, herbicides and insecticides, and cement from entering Site soils and adjacent water bodies.

D. No materials, wastes, effluent, trash, garbage, oil, grease, chemicals, etc., shall be stored in areas adjacent to Unnamed Ditch, site perimeter ditches, Finley Creek, or other waterways. If any waste material is dumped in an unauthorized area, the Contractor shall remove the material and restore the area to the condition of the adjacent undisturbed area.

3.03 MAINTENANCE

A. The Contractor shall dispose of discarded debris from any source whatsoever, in accordance with local and federal regulations.

B. The Contractor shall frequently remove materials no longer required on the site, such as excess excavated material, forms, temporary structures and similar materials and equipment so that, at all times, the site, access routes to the site and any other areas disturbed by site operations shall present a neat, orderly, workmanlike appearance. Contractor is responsible for cleaning and maintenance of all subcontractor staging areas.

C. Before final payment, the Contractor shall remove all surplus material, false work, temporary structures, including foundations thereof, and debris of every nature resulting from his operations, and put the site in a neat, orderly condition; and restore all areas which have been used or disturbed by his operations, to their

original condition or to condition satisfactory to and approved by the Trust's Engineer.

3.04 EROSION CONTROL

A. Unless otherwise permitted by the Agencies having jurisdiction, control measures, in the absence of other locally established limitation, must be adequate to assure that turbidity in the receiving water due to the runoff of silt and clay will not be increased to more than 25 nephelometric turbidity units (NTu) above normal for the condition prevailing.

B. The banks of Finley Creek and Unnamed Ditch are to be protected during construction.

C. Earthmoving vehicles, construction ponds, or pumps shall not discharge any petroleum product or accumulated sediment to Finley Creek or Unnamed Ditch.

D. Silt fencing will be maintained along the banks of Unnamed Ditch and site Diversion Channels located adjacent to the SVE trench and thin barrier curtain wall alignments.

1. The filter fabric shall be fastened to the silt fence posts at the top, center, and bottom with staples. The filter fabric and fence shall bury at least four inches below grade and shall extend at least two feet above grade.

2. Replace silt fence as needed during construction to ensure adequate protection. Remove excess soils in front of silt fence, as needed, during construction to ensure adequate protection.

E. Erosion control measures shall be maintained until grass growth is established.

3.05 DUST CONTROL

A. The Contractor shall keep all excavations, embankments, stockpiles, haul roads, permanent access roads, temporary waste staging areas, and all other work areas within the project boundaries free from dust that would cause a hazard or nuisance to others or contaminate surface water.

B. The Contractor shall, at his expense, keep dust under control at all times on all roadways, and other areas adjacent to the work or on the site of the work including after working hours, Saturdays, Sundays and holidays. Contractor assumes responsibility for any Contract delays or work stoppages due to inappropriate dust control measures.

C. Approved temporary methods of stabilization consisting of motor sweepers, vacuums, spraying water, and a combination of these methods, will be permitted to control dust.

1. Spraying water shall be applied at such intervals as to keep all parts of the disturbed area at least damp at all times, and the Contractor shall have sufficient suitable equipment on the job to accomplish this. Dust control shall be performed whenever a dust nuisance or hazard occurs.

2. Water shall not be salty or brackish; water shall be free of oil, acid and injurious alkali or vegetative matter.

3. Calcium chloride shall not be used.

4. Bankert Pond water may be used for dust control if there is an agreement between the Trust and the Property Owner concerning such usage.

D. All areas undergoing excavation, grading, filling and cutting are subject to dust-inhibiting practices. The use of liquid palliative and penetrating asphaltic materials will not be permitted. Dust-inhibiting practices shall be applied to non-traffic areas subject to blowing as temporary treatment.

3.06 NOISE CONTROL

A. The Contractor shall use every effort and means possible to minimize or eliminate noise caused by his operation which the Trust's Engineer may consider objectionable. The Contractor shall provide working machinery, designed to operate with the least possible noise. Heavy equipment shall not be operated before 7:00 A.M. or later than 9:00 P.M.

3.07 PROHIBITED CONSTRUCTION PROCEDURES

A. The Contractor is advised that the disposal of excess material in wetlands, diversion ditches, stream corridors, and floodplain is strictly prohibited. Any violation of this restriction by the Contractor or any person employed by him will be brought to the immediate attention of the responsible regulatory agencies, with a request that appropriate action be taken against the offending parties. Therefore, the Contractor will be required to remove the fill at his own expense and restore the area impacted.

B. The Contractor shall comply with the following requirements regarding prohibited construction procedures as follows:

1. Filling of wetlands or floodplain areas beyond the location and elevations identified.

2. Indiscriminate, arbitrary or capricious operations of equipment in any stream corridors, any wetlands or surface waters.

3. Pumping of silt-laden water from trenches or other excavations into any surface waters, any stream corridors or any wetlands.

4. Damaging vegetation adjacent to, or outside of, the identified work area.

5. Disposal of trees, brush and other debris in any stream corridors, any wetlands, any surface waters, or at unspecified locations.

6. Open burning of project debris.

7. Location of storage stockpile areas in environmentally sensitive areas.

END OF SECTION

SECTION 01510

TEMPORARY FACILITIES

SECTION 01510

TEMPORARY FACILITIES

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Contractor shall be responsible for arranging and providing all construction facilities and temporary controls as specified herein and as required for the proper and expeditious prosecution of the Work. The Contractor shall pay all costs for such facilities and controls, unless otherwise specified, until such facilities and controls are removed from the site. Construction facilities and temporary controls shall include the following activities:

1. Providing temporary sanitary facilities during the course of the site operations for all site personnel.

2. Establishing equipment and materials staging, storage and laydown areas.

3. Implementing appropriate engineering controls to mitigate migration of potentially contaminated materials and/or liquids from the site during the site remediation operations.

4. Providing field office facilities for Contractor, Trust's as required, and necessary Contractor storage facilities. Contractor is responsible for arranging their own communications, electricity, and all other required utilities.

5. Providing fire protection, lighting, safety equipment, and equipment decontamination as required by OSHA, local regulations, and the specifications herein.

B. The Contractor shall obtain all required permits and connections for temporary utility service. The National Electrical Code and all Local, State and Federal codes, laws, and regulations remain in full effect.

1.02 RELATED WORK NOT INCLUDED

A. Section 01710 – Cleaning

1.03 SUBMITTALS

A. None.

1.04 ACCESS ROADS

A. Temporary driveway(s) for construction traffic shall be as shown on the Design Drawings and/or as approved by the Trust's Engineer.

B. Provide means of removing mud from vehicle wheels before entering streets.

1.05 PARKING

A. The Contractor shall locate suitable parking areas on the site or adjacent to the site as shown on the Design Drawings or as approved by the Trust's Engineer.

1.06 PROGRESS CLEANING

A. Maintain areas free of waste materials, debris, and rubbish. Maintain site in a clean and orderly condition.

B. Remove waste materials, debris, and rubbish from the site, as needed, and dispose of off site.

1.07 REMOVAL OF UTILITIES, FACILITIES, AND CONTROLS

A. Remove portions of all SVE risers piping, and dewatering wells within 2 feet of the surface upon approved completion of the work at the end of Phase I Monitoring.

B. Restore permanent facilities used during construction to specified condition. The Contractor shall be responsible for all damages to properties inside or outside of the ECC Site shown on the Design Drawings such as structures, fences, walls, and lighting, etc., that may result from his operations and shall restore same to their original condition a soon as possible and prior to completion of this Contract.

C. Abandon thin barrier curtain wall piezometers and Site monitoring wells in accordance with Indiana regulations upon approved completion of Phase I Monitoring.

1.08 PROTECTION OF PROPERTIES, STRUCTURE, AND UTILITIES

A. Provide and maintain proper protection from all damage resulting from the performance of the work as specified and/or carried out for all properties, structures, underground and overhead utilities which are within or adjacent to the work area. Any damage resulting from lack of proper protection of the Work shall be repaired at the expense of Contractor.

B. Contractor's equipment working in the vicinity of overhead lines shall at all times be grounded to protect persons and property.

C. Caution shall be exercised in the movement of construction equipment and personnel under, around, and over existing lines and structures (including the diversion channels, aboveground piping and other aboveground equipment, and extraction wells) in the immediate work area. If overhead lines are removed, coordination with the adjacent Third Site is required.

D. Wherever in this specification the Contractor is required to provide protection for materials, equipment, property, or completed or partially completed work, it shall be understood that such protection shall include protection from vandalism or other related acts, as well as protection from damage resulting from the performance of the Work.

E. It is the responsibility of the Contractor to contact the owners of the various utilities in the area of the site prior to starting work on this Contract and also during construction, and determine the exact location of any structures, gas or water mains, electric conduits, answer lines, and all service lines the utilities may have at the site of the Contractor's work, so the Contractor may locate and protect them.

F. The Contractor assumes all responsibility and liability for property damage and bodily injury that may result from damaging or disturbing any structures, facilities, water and gas mains or electric conduits.

G. The Contractor shall be responsible for all damage to utilities, structures, power lines, gas, water and drain lines, tanks, sewers, etc., that may result from his operations and shall restore same to their original condition as soon as possible and prior to completion of his Contract.

1.09 *FIRST AID FACILITIES*

A. The Contractor shall provide and maintain first aid equipment in the various work areas, and provide for the treatment of minor injuries to employees. Contractor will not be required to provide such treatment to employees of other Contractors or of Trust's Engineer. Contractor shall designate personnel with qualifications to administer first aid. Contractor shall be responsible for transportation and treatment of employees with injuries in accordance with their Health and Safety Plan.

B. The Contractor shall provide trained and certified personnel to administer Cardio Pulmonary Resuscitation (CPR) in accordance with their Health and Safety Plan.

PART 2 – PRODUCTS

2.01 MATERIALS

A. Contractor's facilities shall be of adequate size and content for his administration of the contract, storage of materials required for installation, and provision for personnel shelter.

B. Equipment required for the personal safety of workers shall be furnished in full compliance with specific safety requirements of local, and federal agencies including OSHA.

2.02 FIELD OFFICES FOR TRUST'S ENGINEER AND USEPA

A. The existing field office trailer can be used for the Trust's Engineer and USEPA's use. Any other office space required shall be supplied, installed, and maintained by the Contractor.

B. Contractor shall provide electrical hookup to the existing office trailer and shall be responsible for all electrical charges.

PART 3 – EXECUTION

3.01 PERFORMANCE

A. Field offices and storage trailer shall be sited in approved locations and properly set up for all anticipated weather conditions.

B. Contractor shall provide sufficient bottled drinking water to all site personnel during construction.

END OF SECTION

SECTION 01710

CLEANING

SECTION 01710

CLEANING

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Contractor shall furnish all labor, materials, and equipment required to maintain ECC Site free from waste, debris, and dirt caused by Augmented Soil Vapor Extraction work.

B. At the completion of work, Contractor shall remove waste materials, rubbish, tools, equipment, machinery, and surplus material. The Contractor shall clean all sight-exposed surfaces; leave Site clean and neat.

1.02 RELATED WORK NOT INCLUDED

A. Section 01510 – Temporary Facilities

PART 2 – PRODUCTS

2.01 MATERIALS

A. Cleaning materials shall only be those recommended by the manufacturer for the surface to be cleaned.

PART 3 – EXECUTION

3.01 DURING CONSTRUCTION AND OPERATION

A. Contractor shall execute cleaning to ensure that treatment buildings, sheds, and grounds are maintained free from accumulations of waste and debris.

B. Contractor shall wet down dry materials and rubbish, as needed, to prevent blowing dust.

C. Contractor shall, as needed, clean the Site, including back blading earthwork areas as necessary to remove ruts and major surface irregularities, dressing stockpiles, and disposing of waste and debris.

1. Contractor shall maintain a solid nonhazardous waste roll off container on site at all times. The roll off shall be disposed at a secure landfill USEPA landfill and replaced when full.

2. Contractor shall remove tracked dirt off of adjacent driveway, and local roads as needed.

D. Contractor shall remove snow and ice from construction surfaces as conditions require and spread sand over ice patches, as needed, to decrease slip hazard.

3.02 FINAL CLEANING

A. Contractor shall clean the Site of rubbish, litter and other foreign substances. Sweep paved areas broom clean; remove stains, spills and other foreign deposits. Remove waste materials from the Site and dispose of in a lawful manner.

B. Contractor shall remove temporary facilities installed for protection of the Work during construction.

END OF SECTION

SECTION 02110

GENERAL SITE PREPARATION

SECTION 02110

GENERAL SITE PREPARATION

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Contractor shall furnish all labor, materials, and equipment required to perform all site preparation as shown on the Design Drawings and specified herein.

1.02 RELATED WORK NOT INCLUDED

A. Section 1502 – Environmental Protection

1.03 SITE PREPARATION

A. Contractor shall locate and label all underground utilities within the site, especially those near the new SVE trenches and buried piping alignments. Contractor shall arrange for power lines located along the alignment to be de-energized during trenching activities in the vicinity, if needed.

B. Install/maintain erosion and sediment controls as required by Section 01502.

C. Clean existing Decon Pad.

D. Level and prepare pad area for additional water storage tank (T-5).

E. Provide connection to electricity for pumps, field instrumentation, and temporary facilities.

F. Add gravel as needed in parking areas, equipment areas and temporary access roads.

G. Clear and grub, as needed, and provide working platform for thin barrier curtain wall installation equipment as shown in the Design Drawings.

PART 2 – PRODUCTS

2.01 FENCING

A. As part of the site decommissioning activities, commercial grade fencing with vertical supports not less than 10 feet apart (including concrete footings for vertical supports) shall be installed east of the completed curtain wall and tied into the

existing fencing. Fencing removed during site preparation may be reinstalled if in good condition.

PART 3 – EXECUTION

3.01 PROTECTION

A. Trees and limbs outside the construction limits shall not be removed without the prior approval of the Trust's Engineer or the Property Owner.

B. Contractor shall trim trees and shrubs not designed for protection where possible to avoid complete removal.

C. Damage outside the ECC Site Area limits shall be corrected at the Contractor's expense.

END OF SECTION

SECTION 02190

OFF-SITE TRANSPORTATION AND DISPOSAL

SECTION 02190

OFF-SITE TRANSPORTATION AND DISPOSAL

PART 1 – GENERAL

1.01 DESCRIPTION

A. Contractor shall furnish all labor, materials, equipment and incidentals necessary for off-site transportation and final disposal of all wastes associated with the Augmented Soil Vapor Extraction work. Subsurface water, monitoring well development and purge water, decontamination water, and waste water generated by the dewatering phase or operation of the augmented SVE system shall be treated at the on-site waste water treatment facility unless alternate disposal methods are approved by the Trust. Site soils shall be handled as outlined in the Design Report.

B. The Contractor shall perform all work to provide off-site transportation and final disposal of excavated soil material, which can not be treated, disposed of or re-used on site.

C. The Contractor shall ensure that all operations for loading and hauling of all waste are in compliance with the Federal and State Departments of Transportation regulations, EPA Hazardous Waste Regulation 40 CFR Parts 262 and 263, and revised procedures for Implementing Off-site Response Actions (EPA OSWER Directive Number 9834.11, November 13, 1987), and all state and local requirements.

D. The Contractor shall be responsible for acquiring all necessary approvals, registration, and keeping appropriate records of all analysis and disposal records.

1.02 RELATED WORK NOT INCLUDED

A. Section 01460 – Spill Control

B. Section 02110 – General Site Preparation

1.03 SUBMITTALS

A. Off-site Disposal Plan, if off-site disposal is required.

B. Final report documenting quantity of waste disposed and copies of analysis data.

C. Copies of all waste disposal manifests or bills of lading.

1.04 OFF-SITE DISPOSAL PLAN

A. If materials are to be disposed off site, the Contractor shall prepare and submit an Off-site Disposal Plan to the Trust's Engineer at least 5 days prior to handling the material to be disposed.

B. The Off-site Disposal Plan shall include, at a minimum, information on where the contaminated materials removed from the Site will be taken and procedures for handling the wastes. The Contractor shall identify the off-site transporters for the project and waste generator and manifesting procedures in the Off-site Disposal Plan. Only licensed transporters and storage and disposal facilities (TSDFs) shall be used. Soils from ECC have been handled as listed hazardous waste in the past. The plan shall include waste transportation company insurance coverage certification/information.

1.05 VEHICLE REQUIREMENTS

A. Transporter will provide placards and/or identification number as required in USDOT regulations.

B. Transporters of hazardous waste off-site shall be in full compliance with 40 CFR 263 as well as other applicable laws and regulations, including USDOT requirements.

C. Transporters of Special or Industrial waste off-site shall be in full compliance with IDEM waste regulations.

1.06 MATERIAL TO BE ENCOUNTERED FOR POSSIBLE DISPOSAL

A. Site soils

B. General construction debris and trash

C. Personal protective equipment

D. Spent granular activated carbon

E. Excess biopolymer slurry

F. Miscellaneous cleaning/decontamination residues

PART 2 – PRODUCTS – Not Applicable

PART 3 – EXECUTION

3.01 GENERAL

A. Contractor shall organize and maintain the material shipment records/manifests required by the Federal Resource Conservation and Recovery act (RCRA) (Public Law 94-580), the State of Indiana and the State where the disposal facility is located. Contractor shall arrange for manifest signatures with the ECC Trust.

B. Contractor shall coordinate the schedule for truck arrival and material deliveries at the disposal site to meet the approved project schedule. The schedule shall be compatible with the availability of equipment and personnel for material handling operations.

C. Contractor shall use a certified scale, calibrated truck scale with ticket printer and digital weight indicator for weighing trucks prior to or upon delivery at a disposal facility. The scale shall be either a single axle or full truck scale. The facility shall meet the applicable requirements of the National Institute of Standards and Technology Handbook 44 for commercial weighing.

D. During construction phase of the project, all vehicles encountering subsurface soil or ground water shall be decontaminated prior to leaving the Site. Contractor shall inspect all vehicles leaving the Site to ensure than no excess soil adheres to its wheels or undercarriage. All excess soil shall be removed at the decontamination pad.

E. Construction debris, contaminated soil, liquid wastes and other materials to be disposed off-site shall be sampled and submitted to the laboratory for analysis as required by the disposal facilities, and placed in appropriate containers for disposal. Any sampling and/or analysis fees shall be the responsibility of the Contractor.

3.02 HAULING

A. Contractor shall not deliver waste to any facility other than the disposal facility(ies) listed on the shipping manifest or waste disposal notification forms.

B. Contractor shall be responsible for any and all actions necessary to remedy a situation involving material spill in transit or mud and dust tracked off site. This cleanup shall be accomplished at the Contractor's expense.

C. Contractor shall be held responsible for the access routes for road condition, overhead clearance, and weight restrictions used by the hauling Contractor.

D. Contractor shall ensure that trucks are protected against contamination by properly covering and lining them with compatible material or by decontaminating them prior to any use other than hauling contaminated materials.

E. The Contractor shall only use the transporter(s) identified in the Contractor's Off-site Disposal Plan for the performance of work. Any use of substitute or additional transporters must have previous approval from the Trust's Engineer.

F. All contaminated soil, liquid, sludge, and debris shall be transported in water tight, covered containers in conformance with USDOT regulations, and any other applicable regulations.

3.03 OFF-SITE DISPOSAL

A. Contractor shall use only permitted disposal facility(ies). Substitutions or additions shall not be permitted without prior written approval from the Trust's representative, and if approved, shall be at no extra cost to the Trust.

B. Contractor shall be responsible for acceptance of the specific material at an appropriately permitted disposal facility, for ensuring that the facility is properly permitted to accept the stated material, and that the facility provides the stated disposal services.

C. In the case of all disposal facilities, if the identified and approved facility ceases to accept the stated materials or the facility ceases operation, it is the Contractor's responsibility to locate an alternate approved and permitted facility for accepting materials. Contractor is responsible for making the necessary arrangements to utilize the facility, and the alternate facility must be approved in writing by the Trust's Engineer. This shall be done with no extra cost to the Trust.

3.04 RECORD KEEPING

A. Contractor shall obtain the manifest or required waste disposal notification forms, obtain material code numbers, and complete the shipment manifest records as required by the appropriate regulatory agencies for verifying the material type (Code No.) and quantity of each load in unit of volume and weight.

B. Contractor shall prepare a report with written documentation and records verifying receipt/waste disposal including the quantity received of each load at each disposal facility and verification of proper disposal.

END OF SECTION

SECTION 02200

EXCAVATION, BACKFILL, AND COMPACTION

SECTION 02200

EXCAVATION, BACKFILL, AND COMPACTION

PART 1 – GENERAL

1.01 DESCRIPTION

A. Furnish all labor, materials, equipment and incidentals necessary to perform all excavation, backfill, and compaction required to complete the work shown on the Design Drawings.

1.02 RELATED WORK NOT INCLUDED

A. Section 01050 – Grades, Lines, and Levels

B. Section 02110 – General Site Preparation

C. Section 02210 – Augmented SVE Trench Construction

D. Section 02206 – Permeable Reactive Gate System

E. Section 02730 – Vaults and Manholes

1.03 SUBMITTALS

A. Material Source: Name of material supplier for each material utilized on site at least 3 days prior to installation.

B. Certified laboratory testing reports for all imported soil and aggregate no later than 96 hours after the field samples are taken:

1. Grain size analyses showing gradation curve, percent passing for each sieve designation and gradation parameters.

2. When required, moisture-density relationship test (ASTM D1557) results including density versus moisture content curve.

C. As-built drawings of earthwork including copies of backfill and delivery tickets.

1.04 DEFINITIONS

A. Cohesionless and Cohesive Materials

Cohesionless materials include materials classified in ASTM D2487 as GW, GP, SW, and SP. Cohesive materials include materials classified as GC, SC, ML, CL, MH, and CH. Material classified as GM and SM shall be identified as Cohesionless only when the fines are non-plastic (i.e., a plasticity index less than zero). Testing required for classifying materials shall be in accordance with ASTM D4318 and D422.

B. Compaction

The process of mechanically stabilizing a material by increasing its density at a controlled moisture condition. "Degree of Compaction" is expressed as a percentage of the maximum density obtained by the test procedure described in ASTM D1557 for general soil types.

C. Excavation

Removal of soil, rock, hard materials, construction material or debris to obtain a specified depth or elevation.

D. Fill

Specified material placed at specified degree of compaction to obtain an indicated grade or elevation.

E. Rock

Solid homogeneous interlocking crystalline material with firmly cemented, laminated, or foliated masses or conglomerate deposits, neither of which can be removed without systematic drilling and blasting, drilling and the use of expansion jacks, or the use of backhoe-mounted pneumatic hole punchers or rock breakers; also large boulders, buried masonry, or concrete other than pavement exceeding 1/2-cubic yard in volume. Removal of unyielding material will not be considered rock excavation because of intermittent drilling and blasting that is performed merely to increase production.

F. Satisfactory Material

Satisfactory materials shall consist of any materials classified by ASTM D2487 as, GW, GM, GC, SC, SW, CL, and CH.

G. Subgrade

Material in excavation cuts or embankment fills immediately below any sub-base, pavement, backfill, or other improvement. Also, as a secondary definition, the level below which work is referenced.

H. Unsatisfactory Materials

Unsatisfactory materials except for topsoil shall be materials that do not comply with the requirements for satisfactory materials. Unsatisfactory materials include, but are not limited to those materials containing roots and other organic matter, trash debris, frozen materials and stones larger than 3 inches, and materials classified in ASTM D2487 as PT, OH, and OL. Unsatisfactory materials also include man-made fills, refuse, or backfills from previous construction.

I. Instable Material

Unstable material shall consist of materials too wet to properly support the utility pipe, conduit or appurtenant structure.

J. Unyielding Material

Unyielding material shall consist of gravelly soils with stones greater than 3 inches in any dimension or as defined by the pipe manufacturer, whichever is smaller.

1.05 REFERENCES

A.	ASTM D422	Particle-Size Analysis of Soils
B.	ASTM D698	Moisture-Density Relations of Soils and Soil-Aggregate Mixtures Using 5-pound (2.27-kg) Rammer and 12-inch (305-mm) Drop
C.	ASTM D1557	Moisture-Density Relations of Soils and Soil-Aggregate Mixtures Using 10-pound (4.54-kg) Rammer and 18-inch (457-mm) Drop
D.	ASTM D2487	Classification of Soils for Engineering Purposes
E.	ASTM D2922	Density of Soil and Soil-Aggregate In Place by Nuclear Methods (Shallow Depth)
F.	ASTM D2974	Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

G. ASTM D 3017 Moisture Content of Soil and Soil-Aggregate In Place by Nuclear Methods (Shallow Depth)

1.06 SITE CONDITIONS

A. Existing grades and other site information shown on the applicable Design Drawings are approximate.

B. Contractor is responsible for any conclusions drawn from the available borings, cone penetration testing results, and chemical analyses regarding the character of the materials to be expected during the work. The proportions and character of the various materials indicated on the borings and testing results may vary from actual Site conditions.

1.07 SAFETY

A. Methods of operation utilized in work related to these specifications shall be such as to provide maximum protection against injury or death to workmen or the public. Requirements of the United States and State of Indiana Occupational Safety and Health Acts as to safety regulations and procedures shall be adhered to for all work covered under these specifications.

B. The Subcontract shall provide and maintain barricades and signs around excavations as required by OSHA codes.

1.08 JOB CONDITIONS

A. All stockpiling shall be performed by the Contractor at no additional expense.

B. Soil, geosynthetic materials, and crushed stone and/or gravel materials shall be supplied by the Contractor from controlled off-site sources as required. All loading, hauling, stockpiling and protection of the stockpiles shall be performed by the Contractor at no additional expense.

1.09 DELIVERY, STORAGE, AND HANDLING

A. Contractor shall deliver, store, and handle soil and aggregates in a manner to prevent contamination, segregation, saturation, or other deterioration. Delivered materials that are out of spec will be removed from the site and replaced with suitable materials at the Contractor's expense.

PART 2 – PRODUCTS

2.01 SOIL AND STONE

A. Subbase Stone

Material shall be composed of hard, tough, free-draining, non-plastic sand and gravel, slag, or crushed stone, free of muddy material, organic matter, rubbish, debris, or other unsuitable material. Subbase stone shall meet the following gradation requirements when tested in accordance with ASTM D422 (INDOT Coarse #8 or 9 stone).

Sieve Destination	Percent Passing (by weight)
3/4	75-95
1/2	40-85
3/8	20-60
4	0-15
200	0-4

B. Pea Gravel Backfill

Pea gravel shall meet the gradation requirement as specified below when tested in accordance with ASTM D422 (INDOT No. 11 Coarse Aggregate or equivalent), and be free of muddy material, organic matter, rubbish, debris, or other unsuitable material.

Sieve Destination	Percent Passing (by weight)
1/2 inch	100
3/8 inch	75-95
No. 4	10-30
No. 8	0-10

C. Dense Graded (Driveway) Stone

Driveway stone shall be obtained from rock of uniform quality and shall consist of clean, angular fragments of quarried rock, free from soft or disintegrated pieces or other objectionable matter. The stone shall meet the following gradation requirements when tested in accordance with ASTM D422 (INDOT No. 53 Dense Graded Stone, "B" Borrow, or equivalent).

Sieve Destination	Percent Passing (by weight)
1-inch	80-100
3/4-inch	70-90
1/2-inch	55-80
No. 4	35-60
No. 30	12-30
No. 200	5-10

D. Common Fill

For general site grading, and fill, use sand, sandy loam, silty clay loam, sandy clay or silty clay as defined in INDOT 1999 Specification Standards Section 903. Material shall be free from contamination rubbish, debris and other unsuitable materials.

E. Free Draining Gravel (1- to 2-inch diameter)

For SVE trench segment backfill, see gradation in Technical Specification 02210.

PART 3 – EXECUTION

3.01 EXCAVATION – GENERAL

A. Excavation shall be performed to the lines and grades indicated on the Design Drawings or as required to complete the work. During excavation, material satisfactory for backfilling shall be stockpiled in an orderly manner.

B. Excavated material not required or not satisfactory for backfill and contaminated excavated soil shall be handled as described in the Design Report.

C. Grading shall be done as may be necessary to prevent surface water from flowing into site excavations, and any excess water accumulating therein shall be removed to maintain the stability of the bottom and sides of the excavation. Unauthorized over excavation shall be backfilled at no additional cost to the Owner.

D. The Contractor shall dewater the excavation as necessary to perform the required work. All contaminated water shall be collected and transported for on-site treatment.

E. Provide control of dust, at times designated by the Trust's Engineer, by wetting surfaces contributing to the dust problem. Do not use calcium chloride or oils to control dust.

3.02 PIPE and UTILITY TRENCHES

A. Trench walls shall be shored, cut back to a stable slope, or provided with equivalent means of protection for employees who may be exposed to moving ground or cave in as per 29 CFR 1926 regulations and as provided herein. Special attention shall be given to slopes, which may be adversely affected by weather or moisture content.

B. Health and Safety monitoring shall be conducted during excavation of any trenches on the site in accordance with the site safety plan.

C. Bottom Preparation

The bottoms of utility/pipe trenches shall be excavated six inches below required pipe invert and backfilled with pea gravel, or clean sand to provide uniform bearing and support for the bottom quadrant of each section of the pipe.

D. Removal of Unyielding Material

Where unyielding material is encountered in the bottom of the trench (including metal sheeting), such material shall be removed 6 inches below the required grade and replaced with suitable materials as provided herein.

E. Removal of Unstable Material

Where unstable material is encountered in the bottom of the trench, such material shall be removed to the depth directed by the Trust's Engineer and replaced to the proper grade with select granular material as provided herein. This material shall be placed by the Contractor without additional cost to the Trust.

3.03 EXCAVATION FOR APPURTENANCES

A. Excavation for manholes, vaults, collection tanks, sumps, or similar structures shall be sufficient to leave at least 12 inches clear between the outer structure surfaces and the face of the excavation or support members. Removal of unstable material shall be as specified above.

B. When concrete or masonry is to be placed in an excavated area, special care shall be taken not to disturb the bottom of the excavation. Excavation to the final grade level shall not be made until just before the concrete or masonry is to be placed.

3.04 EXCAVATION AROUND EXISTING UTILITIES AND STRUCTURES

A. Excavation around existing underground utilities shall be conducted to prevent compromising the structural integrity of the utilities. Adequate shoring and/or support shall be provided to prevent movement during the trenching operations.

B. Following completion of the excavation operations, backfilling and compaction shall be conducted in accordance with this Section. The Contractor shall be responsible for repairing and/ or replacing any underground utility that is damaged during the site operations at no additional cost to the Trust.

C. An attempt has been made to locate existing structures on the drawings, but the completeness or accuracy of the information given is not guaranteed.

D. If site work approaches pipe, manholes, or other underground structures, digging by machinery shall be discontinued and the excavation shall be done by means of hand tools or as appropriate for safe conditions. Such manual excavation when incidental to normal excavation shall be included in the work to be done under items involving normal site work.

E. Where determination of the exact location of a pipe or other underground structures is necessary for doing the work properly, the Contractor shall provide survey work at his expense.

3.05 STOCKPILES

A. Stockpiles of satisfactory and waste materials shall be placed and graded as specified. Stockpiles shall be kept in a neat and well-drained condition, giving due consideration to drainage at all times.

1. Excavated satisfactory and unsatisfactory material shall be separately stockpiled. Stockpiles of satisfactory materials shall be protected from contamination, which may destroy the quality and fitness of the stockpiled material.

2. Stockpiles shall be covered with plastic and/ or surrounded by silt fence to prevent migration of fine particulates. If any construction material becomes unsatisfactory, such material shall be removed and replaced with satisfactory material from approved sources at no additional cost to the Trust.

3.06 BACKFILLING

A. Backfill material shall consist of satisfactory material as specified. Compaction shall, unless otherwise specified, conform to the requirements of this paragraph. Except for pea gravel and the 1- to 2-inch diameter gravel backfill placed in the SVE trenches, the backfill shall be placed in layers not exceeding 9 inches loose thickness unless otherwise specified. Hand operated compaction equipment shall be used directly around underground utilities.

B. SVE trenches shall be backfilled with 1- to 2-inch size free draining gravel as specified in Specification 02210 to 2 feet below the ground surface.

C. For foundation excavations (manholes), unstable material removed from the bottom of the trench or excavation may be replaced with granular materials placed in layers not exceeding 6 inches loose thickness.

D. Pipe bedding shall be placed and compacted to a height specified on the drawings above the pipe or conduit. The backfill shall be brought up evenly on both sides of the pipe for the full length of the pipe.

E. Before compaction, Contractor may moisten or aerate each lift, as appropriate, to achieve the sufficient compaction.

F. After any manholes, vaults, collection tanks, sumps, or similar structure has been constructed and the concrete has been allowed to cure adequately to support backfill operations, backfill shall be placed in such a manner that the structure will not be damaged by the shock of falling earth. The backfill material shall be deposited and compacted as specified for final backfill, and shall be brought up evenly on all sides of the structure to prevent eccentric loading and excessive stress.

3.07 COMPACTION REQUIREMENTS

A. Compaction is expressed as a percentage of the maximum density. Contractor is required to meet the specified maximum densities for each soil and/or condition.

B. Moisture Requirements

1. Contractor shall provide moisture control to the extent that the soil mix remains in a workable state during placement.

2. Where subgrade or layer of soil requires water prior to compaction, apply water uniformly to surface of subgrade or soil layer as such a rate as to avoid free water from appearing on surface.

3. Remove soil that is too wet to permit compaction to the specified density. Excessively wet soil that has been removed may be stockpiled or spread and allowed to dry.

C. Materials to be compacted to the requirements below:

Material	Required Percent of Max. Dry Density (ASTM D1557)
Sand Bedding	90%
Common Fill for trench cap or Grassed/Landscaped areas	80%
Subbase Stone	90%
Pea Gravel	NA
1- to 2-inch free draining stone	NA

3.08 FIELD QUALITY CONTROL

A. Testing will be performed by the Contractor. The Contractor shall include allowances in his cost and schedule for the necessary quality control measurements, sampling, testing, and evaluation performed to approve the progress of the work. The Contractor shall provide a minimum of 36 hours notice to the Trust's Engineer, during the work day, for work that requires a Trust's Engineer's review or approval.

B. Field Density and Moisture Tests: Test will be performed in sufficient numbers to ensure that the specified density is being obtained.

1. One moisture density relationship will be determined for each subbase or pavement stone material used. Field in-place density will be determined in accordance with ASTM D698 or ASTM D2922. ASTM D2922 results in a wet unit weight of soil and when using this method, ASTM D3017 will be used to determine the moisture content of the soil.

2. A minimum of one field density test will be performed per lift of aggregate base or subbase where requested by the Trust's engineer but not more than every 1,000 square. Field in-place density will be determined in accordance with ASTM D698, ASTM D2922, or other method approved by the Trust's Engineer.

3. The calibration curves furnished with the moisture gages will be checked along with density calibration checks as described in ASTM D3017. The calibration checks of both the density and moisture gages will be made at the beginning of a job, and on each different type of material encountered.

4. Backfill improperly compacted shall be reopened to the depth directed, then refilled and compacted to the density specified at no additional cost to the Trust.

C. Documentation

1. Contractor shall maintain daily logs of measurements of lifts, soil characteristics, and other observations.

2. Contractor shall include results of all moisture-density relationship test (when required) results including density versus moisture content curve, and field density and moisture content tests (ASTM D2922, D3017).

3. Contractor shall submit all field test data to the Trust's Engineer at the completion of the construction of the component.

3.09 EXCAVATION SUPPORT SYSTEM

A. Provide all bracing, sheeting and shoring, where necessary to retain the sides of excavations and to prevent movement or settlement of adjacent structures, utilities, piping, conduit, roads and streets, etc. The Contractor shall be entirely responsible for the strength and adequacy of all such bracing, sheeting and shoring. If the Contractor determines that excavation support is necessary, it shall submit an excavation support plan with the pre-construction submittals. The Contractor is solely and entirely responsible for the safety and support of such structures, utilities, etc., and is liable for any damage or injury caused by or resulting from any collapse, movement, or settlement.

B. All bracing, sheeting and shoring shall be installed, as specified, by the Contractor's structural engineer. The Contractor and its structural engineer are solely responsible for the strength and adequate performance of all sheeting, bracing and shoring included in the work.

C. In no case will bracing be permitted against pipes or other structures in trenches or other excavations.

D. Contractor shall keep all bracing, sheeting and shoring in-place and functioning as designated, until replaced by permanent construction.

E. When no longer required, Contractor shall remove all bracing, sheeting and shoring except that which was specifically designed, to remain as part of the permanent structure.

3.10 GRADING

A. General

Contractor shall uniformly grade areas within limits of grading under this Section, including adjacent transition areas. Contractor shall smooth finished surface within specified tolerances, compact uniform levels or slopes between points where elevations are indicated or between such points and existing grades.

B. Contractor shall grade areas adjacent to building lines to drain away from structures and to prevent ponding. Contractor shall finish surfaces free from irregular surface changes.

C. Compaction

After grading, compact subgrade surfaces to the depth and indicated percentage of maximum density for each material classification.

END OF SECTION

SECTION 02206

PERMEABLE REACTIVE GATE SYSTEM

SECTION 02206

PERMEABLE REACTIVE GATE SYSTEM

PART 1 – GENERAL

1.01 DESCRIPTION

A. The Site, as shown on the Design Drawings.

B. The Contractor shall provide all supervision, labor, materials, equipment, tools, instruments, and supplies to construct the Permeable Reactive Gate System (PRGS) as specified herein and as modified, if needed.

C. The 4-inch diameter PRGS conveyance pipes that exit each pipe connection manhole connects SVE Trench Segments 1, 2, 3, 4, and 5 into one pipe and from Trench Segments 6 and 7 into a second pipe. The two PRGS conveyance pipes shall be sloped toward the PRGS collection manhole, where the accumulated water will be pumped to an underground fully contained water-tight, fabricated concrete flow-through treatment vessel. The treatment vessel shall contain granular iron to treat water that flows from the SVE trench segments following completion of the Active Phase. The treated water shall flow out of the treatment vessel through an outflow pipe, to Unnamed Ditch.

D. Contractor shall construct the treatment vessel such that the final elevations of the inlet and outlet pipes in the treatment vessel preclude the potential for a siphon to form, which could remove water from the treatment vessel. The granular iron and iron/sand zone within the reactive vessel must remain saturated at all times for optimal performance of the PRGS. Contractor may schedule installation of granular iron materials for the end of the Active Phase.

1.02 RELATED SECTIONS

A. Section 15050 – Piping

B. Section 02730 – Vaults and Manholes

C. Section 02200 – Excavation, Backfill, and Compaction

D. Section 02280 – Geotextiles

1.03 QUALITY CONTROL

A. All PRGS installation activities shall be conducted by a Contractor that has experience in the installation of PRGSs.

B. Granular Iron

1. Grain size sieve analyses are to be completed on representative samples collected at specified intervals during construction or acceptance of the material, in accordance with ASTM D422, Particle-Size Analysis of Soils (only the sieve portion of the test has to be completed). The results are to be within the range specified in Table 1.

2. All involved parties should reserve the right to visit the manufacturer during the production run to visually inspect the manufacturing process and collect random samples at that time. The manufacturer should provide reasonable assistance to obtain these samples.

C. Sand

1. Grain size sieve analyses are to be completed on representative samples collected at specified intervals during construction or acceptance of the sand, in accordance with ASTM D422, Particle-Size Analysis of Soils (only the sieve portion of the test has to be completed). The results are to be within the range specified in Table 1.

Table 1: Granular Iron and Sand Gradation Requirements

Sieve Size		**Weight Percent Passing**
US Standard Mesh Number	**mm**	
8 (passing)	2.0	95–100
16	1.2	75–90
30	0.6	25–45
50	0.3	0–10
100 (retained)	0.15	0–5

D. Granular Iron/Sand Mixture

1. The granular iron and sand shall be mixed into a uniform mixture using suitable equipment. Satisfactory means, incorporating weighing, or metering shall be provided to assure the proper ratio of sand to granular iron is maintained. Equipment such as concrete trucks, mobile concrete mixers (e.g., Elkin Mixer), or stationary concrete mixers (e.g., pug mills) are suitable alternatives.

2. All equipment should be clean of foreign materials (e.g., cement mix, soil, stones, etc.).

3. The granular iron/sand mixture shall contain 10 percent iron by weight dry.

4. The weights of sand and iron mixed in each batch shall be measured with approved methods and recorded.

5. The sand and granular iron shall be mixed to provide a uniform mixture. The uniformity of the mixture shall be determined with a magnetic granular iron separation test as described in Part 4. The results of the magnetic separation test must be within a specific tolerance of the specified ratio.

6. If the iron/sand mixing is to occur off site, the mixing contractor must be made aware of iron handling and storage issues (i.e., keeping it covered and dry). The granular iron/sand mixture must be stored in a manner similar to the granular iron.

7. The granular iron and sand mixture may be stored prior to installation. The maximum storage times should be based on the moisture content of the material, as shown in Table 2. Storage for an extended period may result in oxidation of the iron.

8. During transport and handling, care should be taken to minimize vertical drop and vibration of the finished product to prevent separation/segregation.

9. The percent granular iron on a dry weight basis is to be determined with the Magnetic Separation Test as described in Part 4. The testing should be completed on representative samples collected from the mixing device. The frequency of testing will depend on the potential variability of the mixing method. Typically more testing is completed in the early stages of the mixing and this frequency is reduced if the results are adequate. The percent granular iron must meet a minimum percent specified on a dry weight basis for the project.

10. Where the granular iron and sand is mixed in a batch process, the percent granular iron can be determined on a bulk basis. The mass of sand is to be determined by an approved method such as a truck scale, portable scale or bucket scale. The mass of wet sand must be corrected for moisture content, either with the moisture content determined with the magnetic separation test or a moisture content test on the sand. This provides assurance that the correct tonnage of granular iron is emplaced.

11. The granular iron sand mixture shall not be stored for periods greater than specified in Table 2.

Table 2: Iron/Sand Maximum Storage Times

Iron/Sand Mixture Moisture Content (Weight Percent)	Iron/Sand Mixture Maximum Storage Times After Mixing (hr)
0 to 3	72
3 to 6	48
6 to 9	24
Greater than 9	8

PART 2 – PRODUCTS

2.01 MATERIALS

A. Granular Iron Specifications

1. The granular iron should consist of approved dry material free from contamination oils, greases, or other foreign organic substances.

2. The granular iron shall be:

   Connelly-GPM Inc.
   3154 South California Avenue
   Chicago, Illinois
   Telephone: 773-247-7231
   Fax: 773-247-7239
   Product: ETI-CC1004 (-8 to +50 US Standard Mesh Size), or approved equivalent.

3. The gradation of granular iron should approximate the range specified in Table 1, and be approved.

4. Granular iron should be transported and arrive on site at near ambient temperatures.

5. The granular iron unloaded at site should be protected from contact with water at all times. Stored granular iron should be covered with impermeable sheets anchored or tied in place, if stored outdoors. Granular iron should not be stored directly on the ground surface.

6. Protective packaging should not be removed from granular iron until final placement in the PRGS vessel or mixing with sand. Unused portions of granular iron shall be returned to storage and protected in accordance with the above requirements.

B. Sand Materials for Reactive Mixture

1. The gradation of the sand shall approximate the grain size range specified in Table 1 and be similar in mineralogy to the native aquifer materials, and be approved.

2. Sand shall be free of stones, clay particles, debris, organic matter, and other foreign material.

3. Sand should be in as dry a condition as possible prior to mixing with the granular iron.

C. PRGS Treatment Vessel

Contractor shall supply and install a treatment vessel as shown in the Design Drawings. The vessel shall be 13 feet wide by 17 feet long and approximately 9.5 feet deep. Actual depth will depend on the final elevation of the ground surface. The vessel shall have the vertical, water-tight divider walls, each with an opening for one 4-inch ID, Schedule 40 PVC pipe. Walls of the vessel shall be reinforced as appropriate (reinforcing design to be determined by Contractor) and coated with a waterproofing sealer/mastic. The vessel shall be placed on stable soils or a minimum 12-inch layer of compacted granular bedding. Hatch door(s) shall allow access to all three chambers and shall include venting, which prevents rain and surface water infiltration.

The 4-inch PVC pipe shall be placed at the base of the pretreatment and treatment chambers of the vessel and shall be slotted except for the last 1 foot and caulked at the divider penetration to prevent leakage between chambers. The slotted section shall be wrapped with a porous non-woven geotextile and surrounded by sand (same sand sizes as listed in Table 1) prior to placement of the pretreatment and treatment media. Vertical sections of the pipes in the vessel shall be installed within 0.05 feet of the elevations shown on the design drawings. Any alterations to the vessel configuration must be submitted as a PRGS Construction Submittal for review and approval.

PART 3 – EXECUTION

3.01 PERMEABLE REACTIVE GATE SYSTEM

A. All PRGS conveyance piping shall be constructed of 4-inch I.D. Schedule 40 PVC pipe. PRGS conveyance pipes 1 and 2 shall flow into a PRGS collection manhole located in the area between SVE Trench Segments 5 and 6.

B. Treatment of the till water will be performed within a treatment vessel filled with a mixture of granular iron and silica sand installed on the south end of the site. The

combined PRGS water shall exit the PRGS collection via a 1.5-inch diameter PVC pipe from the pump installed in the PRGS collection manhole. This piping shall extend to the treatment vessel. The PRGS vessel will consist of a pretreatment zone, a treatment zone, and a discharge chamber. The pretreatment zone at the influent (north) end of the vessels shall be filled with a mix of granular iron and sand. The primary purpose of the sand/iron pretreatment zone shall be to remove dissolved oxygen present from the influent water. The sand/iron mixture shall contain 10 percent iron by dry weight. The treatment zone, consisting of 100 percent iron, shall be placed within the center portion of the vessel. The discharge zone, with no media, shall be placed at the effluent (south) end of the vessel.

C. Based on design calculations, approximately 633 cubic feet of 100 percent granular iron and 64 cubic feet of the sand/iron mixture shall be used in the treatment vessel.

D. The outlet pipe shall be fitted with a sample collection port.

E. The PRGS treatment vessel shall be equipped with a removable lid at the ground surface with an access hatch (e.g., Bilco Type K floor door, www.bilco.com).

F. A low pressure gas vent (i.e., pressure relief valve) shall be installed on the top of the vessel which shall operate at a low pressure (e.g., 5 inches of water column).

G. The effluent pipe (4-inch diameter Schedule 40 PVC) from the vessel shall be sloped to the existing carrier pipe at invert elevation 875 through the thin barrier curtain wall and continue to a discharge point at Unnamed Ditch. A check valve (Tideflex TF-1 type or approved equivalent) shall be placed on the discharge end of the effluent pipe. An outflow structure shall be installed at the discharge point to minimize erosion of the bank of Unnamed Ditch.

3.02 HEALTH AND SAFETY

A. The iron material and silica sand are dust nuisances and adequate personal protective equipment should be worn at all times while handling or being in close proximity to iron material. MSDS data sheets are available from the manufacturer.

B. The contractor should be aware that mixing of iron and sand may cause the temperature of the mixture to increase by several tens of degrees due to friction and oxidation of the granular particles.

PART 4 – TESTING

4.01 MAGNETIC SEPARATION TESTING PROCEDURE

A. Weigh the empty containers that the samples will be collected in.

B. Samples (about 250 to 1,000 g) of the iron-sand mixture are collected from the discharge of the mixing device (e.g., chute of a concrete mixer) and/or from the backfilled material within the excavation. The frequency and location of samples is dependent on the objectives of each project.

C. Weigh the sample (empty container and sample) and record the weight. Determine the net weight of the sample by subtracting the empty sample container weight. A suitable weighing device must be used.

D. Dry the sample. If cemented together during drying, lightly breakup, weigh and record the net weight.

E. Spread the sample out in a suitable container (e.g., bowl, pan, cardboard box, etc.).

F. Cover the magnet in a material (such as a plastic bag) to allow the magnetic material to be easily separated from the magnet.

G. Pass the magnet over the sample to remove the magnetic fraction. Care must be taken to minimize the trapping sand particles within the iron grains. The magnetic fraction is removed from the magnet and placed in a container.

H. Continue passing the magnet over the material until no more magnetic material is removed. Mixing of the non-magnetic fraction between passes may be required to obtain all the magnetic particles.

I. The magnetic fraction may contain some non-magnetic (sand) particles. Repeat Steps 5 to 8 at least three more times to ensure the magnetic and non-magnetic fractions are completely separated. After each separation, the non-magnetic fraction should be added to the non-magnetic fraction from the previous separation.

J. Weight the magnetic and non-magnetic fractions and record weights. The total net weight of the magnetic and non-magnetic fractions should be the same as the weight prior to separation.

K. The dry iron net weight percent is determined by:

$$\text{Dry Iron Net Weight Percent} = \frac{\text{Net Weight of Magnetic Material}}{\text{Total Net Weight of Dry Sample}} \times 100$$

END OF SECTION

SECTION 02210

AUGMENTED SVE TRENCH CONSTRUCTION

SECTION 02210

AUGMENTED SVE TRENCH CONSTRUCTION

PART 1 – GENERAL

1.01 DESCRIPTION

A. The work provided for herein consists of furnishing all plant, labor, equipment, and materials for the construction of the augmented soil vapor extraction (SVE) trenches to be installed by trench method described at the locations and depths shown on the drawings.

B. Trench installation includes excavation under a biopolymer-based viscosifier slurry, backfill with gravel, installation of polyvinyl chloride (PVC) SVE screen, and permeable reactive gate system (PRGS) piping, installation of PVC dewatering wells, and surface backfill. Also included is biopolymer treatment, as required; development by trench flushing, and waste slurry disposal. Seven trench segments are to be constructed.

1.02 RELATED WORK NOT INCLUDED

A. Section 01710 – Cleaning

B. Section 02190 – Off-site Waste Disposal

C. Section 02200 – Excavation, Backfill, and Compaction

1.03 SUBMITTALS

A. Augmented SVE Trench Construction Plan (as described in Section 1.07, including qualifications of Contractor, resume of slurry specialist and biopolymer viscosifier material specifications shall be submitted at least 10 days prior to the Pre-Construction Conference.

B. Supplier laboratory grain size certification for the free-draining 1- to 2-inch diameter gravel shall be submitted at least 10 days prior to start of trench construction.

C. Geomembrane and geotextile manufacturer material certification shall be submitted at least 10 days prior to the start of trench construction.

D. Trench Bottom and Backfill Profiles: Three copies shall be submitted no later than 48 hours after the applicable work or inspection is completed.

1.04 DEFINITIONS

A. Augmented SVE Trenches

For this project, an augmented SVE trench is an approximately 24-inch wide trench excavated in the ground by the slurry method, and backfilled with a free draining gravel to form a high permeable zone for vapor and subsurface water collection.

B. Biopolymer Slurry Method of Excavation

The biopolymer slurry method of excavation is the process of digging a vertical-walled trench, which is supported by keeping the trench full of water thickened with a biopolymer-slurry, based fluid viscosifier. The purpose of the slurry is to support the walls/floor of the trench to prevent the collapse of trench sidewalls prior to backfilling of the trench.

C. Biopolymer-Based Viscosifier

A biopolymer-based viscosifier is a natural polymer manufactured from the Guar Bean or equivalent.

D. Biopolymer Slurry

The slurry is a stable mixture of guar or other biopolymer-based viscosifier and water.

E. Backfill

Free-draining gravel, crushed limestone, or other approved gravel with a nominal size of 1- to 2-inch diameter as specified in Section 2.02.

F. Subsurface Water Level

The subsurface water level is the water level in the till or sand and gravel unit as determined from piezometers and wells.

G. Slurry Trench Specialist

A slurry trench specialist is an individual who has had over 5 years of experience in slurry trench construction and has knowledge in all aspects of slurry trench construction.

H. Quality Assurance Testing

The Trust's Engineer may perform quality assurance testing on the biopolymer-based viscosifier slurry and backfill materials using the laboratory and equipment

furnished by the Contractor. Testing by the Trust's Engineer will in no way relieve the Contractor of the responsibility of performing tests necessary to meet the construction requirements. The Contractor shall make available testing equipment to the Trust's Engineer. All routine quality control testing procedures being conducted by the Contractor shall be available for inspection by the Trust's Engineer at any time.

I. Till Unit

Silty clay and silt soils likely deposited during the glaciation period. The Upper Till is generally in the upper 10 to 20 feet at the ECC Site. The Lower Till is generally found below the saturated sand and gravel unit.

J. Sand and Gravel Unit

The granular soil strata below the upper till and generally found below a depth of 10 to 20 feet at the ECC Site. The sand and gravel unit is mostly saturated, with an upward gradient in some areas.

1.05 QUALIFICATIONS

A. The Contractor shall submit evidence of competence in slurry trench construction, having completed a minimum of three similar projects within the last 5 years.

B. This evidence shall document that the Contractor will have sufficient competent personnel to carry out the operations specified and such personnel shall have experience in this type of construction. A slurry trench specialist shall be employed by the Contractor to control the composition, mixing, placing, cleaning, and maintaining of the slurry and backfill.

C. Credentials of the slurry trench specialist shall be submitted to the Trust's Engineer for approval at least ten days prior to the Preconstruction Conference.

1.06 REFERENCES

A.	ASTM C 143-90	Standard Test Methods for Slump of Hydraulic Cement Concrete.
B.	ASTM D 1140-90	Standard Test Methods for Amount of Material in Soils Finer Than the #200 Sieve.
C.	ASTM D 2217-85	Standard Test Methods for Wet Preparation of Soil Samples for Particle Size Analysis and Determination of Soil Constants.

1.07 AUGMENTED SVE TRENCH CONSTRUCTION PLAN

A. The Augmented SVE Trench Construction Plan shall provide a detailed description of the means and methods used to construct the augmented SVE trenches. At a minimum, it shall include:

1. Equipment and methods used to excavate SVE trenches, including slurry mixing and pumping; treatment and disposal, backfilling, SVE and PRGS pipe installation and dewatering well installation.

2. Safety precautions to be used to safely allow installation of the SVE piping and PRGS conveyance piping in the trench without collapse or water blow-in.

3. Methods to avoid damage to the existing thin barrier curtain wall during construction.

4. Size and type of biopolymer slurry staging areas including slurry tank location, pumps, soil and biopolymer storage, slurry treatment additives, and equipment storage.

5. Augmented SVE trench quality assurance testing program and sample reports.

6. Augmented SVE trench development methods and additional flushing pipes, if necessary.

7. Excess slurry handling (treatment, equipment, and disposal methods).

PART 2 – PRODUCTS

2.01 GENERAL

The slurry for supporting the sides of the trenches shall consist of a stable mixture of biopolymer-based viscosifier and water. At the time of introduction into each trench, the viscosity shall be at least 50 seconds as measured by the Marsh Funnel. If desired, the Contractor may add a weighting agent to increase the density of the slurry.

2.02 MATERIALS

A. Biopolymer-Based Viscosifier and Additives

1. Material shall be processed from the Guar bean or approved equivalent. Material shall be non-toxic and non-polluting.

2. Biopolymer-based viscosifier shall be capable of supporting the walls of the recovery trench when mixed with potable water.

3. Contractor is responsible for preservation of the fluid using sodium hypochlorite, sodium bicarbonate, or other approved additives. Biopolymer-slurry shall be able to be completely broken down through polymer oxidation once trench construction is complete. All potentially clogging degradation by-products shall be flushed from the system prior to acceptance.

4. Representative products and manufacturers include:

   a. G-150 Guar as manufactured by Rantec Corporation, P.O. Box 729, Ranchester, WY, 82839, (307) 655-9565.

   b. Liquid Guar CM as manufactured by Drilling Specialties LLC, 1001 Six Pines Drive, The Woodlands, Texas, (800) 423-3985.

B. *Water*

The Contractor shall supply all water required for mixing with biopolymer slurry to produce slurry. Contractor shall use potable water. There is no on-site water source. The T-4 clean water storage tank (150,000 gallons) may be used for potable water storage.

C. Horizontal SVE Screen

1. The Contractor shall supply 4-inch diameter PVC Schedule 40 screen to be placed within the gravel backfill.

2. The horizontal SVE screen shall be slotted with 0.020-inch slot or equivalent.

3. One 2-inch diameter PVC vertical riser is to be connected to the horizontal SVE screen in each trench segment and will be equipped with a vacuum supply port, valve, flow monitoring port, and vacuum gage (gage range to be determined in the field).

4. A "T" connection and gate valve connecting to 4-inch diameter solid PVC Schedule 40 PRGS conveyance piping shall be included at the PRGS manholes. The solid 4-inch PVC PRGS conveyance piping shall be installed slightly below the horizontal well screen to facilitate flow from the SVE screen.

5. Backfill shall be placed around the SVE screen and related riser and connection piping without damaging or displacing the installed piping.

D. PRGS Conveyance Pipe

1. Contractor shall supply 4-inch diameter Schedule 40 PVC (solid) piping to be placed at certain elevations in the trench backfill, and continue in pipe

trenches to connect to the adjacent trench segment. The PRGS conveyance pipe shall be hydraulically connected to the SVE screen so as to collect subsurface ground water after SVE and dewatering activities are completed. The gate valve between the PRGS conveyance piping and the SVE screen shall be fitted with a valve that can be opened from the ground surface or installed in a manhole that can be entered by the Contractor to open the valve. Granular bentonite shall be added near the pipe at its end trench exit point(s) to minimize the possibility for cross-trench connection.

E. Dewatering Wells

1. Dewatering wells shall include screens that are 4-inch diameter, PVC, Schedule 40, 0.010-inch slot or equivalent. The screens will attach to standard Schedule 40 PVC 4-inch diameter PVC threaded riser pipe to the surface.

2. One dewatering well shall be installed per trench segment during backfilling of the trench.

3. A plug shall be installed at base of well.

4. Contractor may use these wells for biopolymer development, but shall be responsible for adding temporary wells or piping as needed for flushing of the biopolymer slurry, as needed.

F. Backfill

1. Free Draining Gravel

The material for backfilling up to within 2 feet of the ground surface shall be 1- to 2-inch diameter gravel with less than 5 percent fines (< #200 sieve) with ASTM D422 gradation as follows:

Sieve Size	Percent Passing (by weight)
3 inch	90–100
2 inch	0–100
1 inch	0–10
No. 200	0–5

2. Clayey Backfill

The material for backfilling the upper 2 feet (widened section) of the trenches shall be "clean" clayey fill that fits the definition of Common Fill. The material may be imported from an off-site source. Contractor shall provide documentation to demonstrate the fill is "clean." Excavated soils from the upper 2 feet of the trench may be used as backfill if analyzed for VOCs and

SVOCs and no significant contamination is present. Additional thickness of clayey backfill may be added as needed to minimize surface water infiltration into a trench segment.

3. Compacted well graded driveway stone shall be used for the area(s) of roadway crossings as shown on the Design Drawings.

G. Geomembrane/Geotextile

1. Duraskrim (coated geotextile), polypropylene, PVC, or high density polyethylene (HDPE) sheeting, extending 1 foot each side of trench plus the trench width.

2. Geomembrane shall have with minimum burst strength (ASTM-D 751 mod.) of 90 pounds, and a minimum grab tensile strength of 95 pounds.

3. The geomembrane shall be underlain by a non-woven geotextile as described in Specification 02280.

PART 3 – EXECUTION

3.01 EQUIPMENT

A. Contractor shall furnish suitable plant and equipment for excavation of the trenches, mixing and placing slurry, cleaning of bottoms of trenches, for hauling, mixing and placing the backfill material, recirculation of the slurry as required, placing the horizontal SVE/PRGS piping, flushing pipe, dewatering wells and riser pipes, backfill, and surface treatment, biopolymer, and disposal of excess biopolymer slurry.

B. Trenching Equipment

1. The equipment used for excavation of the augmented SVE trenches shall be any type of earthmoving equipment capable of performing the work indicated on the drawings and/or specified herein. Excavating equipment buckets shall be non-perforated, heavy-duty models. Fabrication of the buckets shall be such that raveling of the sides of the trench is minimized and width of the trenches is maintained.

2. The equipment shall be appropriate for excavating a 24-inch wide trenches. Any greater width shall require approval prior to start of excavation. The excavating equipment shall be capable of excavating the width trench in a single pass from the existing ground surface to the depths shown on the Design Drawings (approximately 10 to 15 feet). The equipment width shall not be greater than the existing gravel drive or cause damage to the adjacent remedial area cap or existing thin barrier curtain wall.

3. No blasting will be permitted to remove unyielding soil or rock, if needed. Special rock removal equipment (hoe rams, chisels, drills, etc.) shall be approved prior to use by the Trust's Engineer. Existing sheeting may need to be removed in some areas.

C. Slurry Mixing and Placing Plant

1. The slurry mixing and placing plant shall include a suitable mixer capable of producing a colloidal suspension of biopolymer-based viscosifier in water, a mechanically agitated sump, pumps, valves, hoses, supply lines, and small tools; all as required to mix and provide a continuous supply of slurry to the trench excavation.

2. Slurry shall be stored in aboveground tanks. The use of excavated pits will not be allowed.

D. Cleaning Equipment

1. Equipment for cleaning the slurry shall consist of a plant normally used for this type of operation.

2. Any wash water must be containerized and transported to the ECC wastewater treatment system.

E. Backfill Placing Equipment

1. The free draining gravel backfill shall be placed by the tremie method. Equipment shall be capable of placing the gravel using this method or other approved placement method.

2. The upper soil backfill may be placed with a bulldozer or other equipment needed to achieve the required results. The silty clay backfill shall be compacted using sheeps foot, or equivalent, compaction methods.

3.02 EXCAVATION OF AUGMENTED SVE TRENCHES

A. The seven trench segment locations shall be as indicated on the Design Drawings, approximately 5 feet from the thin barrier curtain wall or as approved in the Augmented SVE Trench Construction Plan. Manholes and PRGS conveyance pipe trenches will be installed as shown in the Design Drawings.

B. The base of the SVE trenches shall be 3 feet from the top of the lower-saturated sand and gravel unit or as shown on the Design Drawings. The Contractor shall immediately notify the Trust's Engineer if the sand and gravel unit is encountered during construction of the trenches.

C. The trench will be excavated to within 0.3 foot of the elevation shown on the drawings, or as directed by the Trust's Engineer. The toe of the slope of the trench excavation shall precede the toe of the backfill slope by a minimum of 10 feet at all times, but not more than 40 feet.

D. The Contractor shall provide a positive means for determining the final bottom elevation of the excavations, and the bottom elevation of the excavations shall at all points meet the approval of the Trust's Engineer. Contractor shall notify the Trust immediately if the sand and gravel unit is penetrated.

E. Excavation at angles in the alignment of a trench shall be made in such a manner as to ensure a continuous full depth and width for the augmented SVE trench segment.

F. Power lines may need to be de-energized and/or removed temporarily during construction near the entrance gate power poles. Contractor shall arrange for the power interruption with the local power company and the operators of the on-site treatment plant. The work shall be scheduled to minimize the time of power interruption to the Site and adjacent Third Site activities.

G. Contractor shall coordinate temporary removal of existing piping, which connects the ECC treatment system and adjacent Third Site. Costs for the pipe disconnect and re-connect will be handled between the ECC Trust and Third Site Trust.

H. Prior to initiating the trenching operations, the Contractor shall locate all underground utilities. If required, limited exploratory excavation operations shall be performed to verify underground utility locations.

I. The slurry within the trench shall be kept free of soil materials until placement of the backfill.

3.03 EXCAVATED MATERIAL

A. Material excavated from the trench may be placed temporarily along the trench. The distance between the trench and the excavated material shall not be less than 10 feet.

B. Excavated material shall be removed and handled as described in Section 2.8 of the Design Report. All stockpiles are restricted to the construction staging area with a liner placed under excavated material so that liquids can drain back into the trench.

3.04 SLURRY PLACEMENT

A. Slurry shall be introduced into the trench at the time trenching is begun. The level of slurry in the trench shall be maintained within 2 feet of the ground surface, or as required for trench stability.

B. Contractor is alerted to the fact that the trenches are designed to terminate at a depth 3 feet above the top of the saturated sand/gravel unit. Excavating into the sand/gravel unit may result in the inability to dewater trench. Soil exploration findings are available for review. Any work resulting from trench collapse, loss of slurry, breach of the sand and gravel unit or stability issues, if necessary, is deemed included in the Contract and it shall be performed at no additional cost to the Trust.

C. Contractor to use pumps and hoses equipment to transfer the slurry from the holding tanks to the augmented SVE trenches. At no time will the Contractor be allowed to transport slurry to the trenches in excavator buckets or shovels.

D. Contractor shall recirculate and treat the slurry as required for maintenance and control of the slurry.

E. Contractor is responsible for containing and cleaning up any spilled slurry.

3.05 DEWATERING WELL INSTALLATION

A. PVC dewatering well screens and riser piping shall be installed (one per trench segment) once the required trench depth has been reached and the trench walls stabilized with the biopolymer mixture. The PVC riser and screen shall be lowered vertically into the trench through the slurry. The backfill shall be placed around the piping to maintain alignment of the casing. The inside of the PVC well materials shall be thoroughly cleaned out prior to installation of pumps, level controls, piping etc.

B. After backfilling, biopolymer slurry removal and trench segment construction is complete, the dewatering wells shall be equipped with Grundfos Redi-Flo3 electric submersible pumps (single-phase 110–115 volts). Each pump will be equipped with Dry-Run Protection, which will automatically shut the pump off when the water level drops below the pump inlet. The pumps in dewatering wells shall be equipped with a minimum 1/3 HP motor. Water level will be controlled by high/low shut offs set with a pressure transducer such as a Druck probe or approved alternate. Power for the pumps and transducers shall be installed by the Contractor from the existing power panel on-site or an additional panel as needed.

The outflow connection for trench segments will consist of 3/4-inch diameter PVC piping. Each pump will be placed 3-inches from the base of dewatering well.

3.06 BACKFILLING THE AUGMENTED SVE TRENCHES

A. The gravel backfill shall be placed in the trench by the tremie method or other method approved by the Trust's Engineer. Slurry may be mixed with the gravel to facilitate placement. The gravel backfill shall be placed from the base of the trench up to 2 feet below the ground surface (after SVE screen and PRGS pipe placement).

B. Slotted SVE screen shall be placed horizontally in the trenches at the elevation(s) shown on the Design Drawings. The horizontal piping shall terminate approximately 3 feet from one end of the trenches and extend into the adjacent manhole at the other end. Place to one side of the trench to allow space for dewatering well riser pipe. Solid PVC riser pipes (one for instrumentation and one for vacuum inlet) shall be connected to the SVE piping as shown on the Design Drawings.

C. PRGS conveyance piping shall run through the trench segments, placed on the gravel backfill (below the SVE screen). A solid or flexible connection between the PRGS conveyance pipe and SVE screen shall be made at the manhole at the lower-end of the trench.

D. The upper 2 feet of each trench (above the gravel) shall be widened an additional 1 foot on each side and a non-woven geotextile then a geomembrane (see Section 2.02) shall be placed above the gravel, extending to the widened portion of the trench. The pieces of geotextile and geomembrane along the trench length shall be overlapped a minimum of 3 feet along the trench alignment.

E. The top 2 feet of the trench (widened section above the geomembrane) shall be backfilled with clean clayey fill. The clayey fill shall be placed in at least three lifts, and each lifted compacted. Excavated soil may be used if previously tested to determine it is not contaminated.

F. At two locations, at least 6 inches of driveway stone shall be placed and compacted at the surface for a roadway crossing. These locations are shown on the Design Drawings.

3.07 AUGMENTED SVE TRENCH DEVELOPMENT

A. The augmented SVE trenches shall be developed by treating the biopolymer slurry to initiate degradation and by flushing of the trench.

B. The Contractor shall initiate the biopolymer slurry degradation process after placement of the free-draining backfill. The Contractor will install temporary PVC wells or flushing pipes at strategic locations in the trenches to allow access to the slurry in the trench. Installation of these temporary wells will be considered part of the work and no additional compensation will be provided for their installation. The dewatering wells and SVE screen may also be used for development.

The subcontractor shall thoroughly flush and develop the augmented SVE trench to achieve silt-free and slurry-free conditions. Approximately two pore volumes of the trench shall be removed during the trench development. The flushing and development shall continue until Biological Oxygen Demand (BOD) decreases to 1,000 mg/l or lower and viscosity measurements by Marsh Funnel method are similar to measurements of the potable water (25 to 30 seconds). Pumps and water

for development shall be supplied by the Contractor. Excess water from the flushing activities will be transported to the ECC water treatment system.

C. The temporary wells (if installed) shall be removed upon completion of trench flushing/development

3.08 SEVERE WEATHER OPERATIONS

A. Excavation of the slurry trench, mixing and placing of slurry, and mixing and placing of backfill will not be allowed when the air temperature is below 30 degrees Fahrenheit or when in the opinion of the Trust's Engineer, severe weather such as snow, rain, or ponding water, may be detrimental to the slurry trench installation or may affect the accuracy of quality control testing.

B. Weather related delays shall not constitute grounds for additional payment or extension of project time for the Contractor.

3.09 CLEANUP AND DISPOSAL OF SLURRY AND EXCAVATED SOIL

A. Excess slurry displaced by the backfill shall be reused or containerized, treated to initiate degradation and transported to the ECC water treatment system.

B. Excavated soil should be characterized and handled as discussed in Section 2.5 of the Design Report.

3.10 QUALITY CONTROL

A. The Contractor shall establish and maintain quality control for all work specified in this section to assure compliance with contract requirements and maintain records of their quality control for all construction operations, including, but not limited to the following:

1. Excavation – Limits, depth, disposition of materials, timeliness and profiles.
2. Materials – Biopolymer and backfill material conform to the specifications.
3. Construction – Conforms to the specifications and drawings.
4. Slurry and Backfill Tests.

B. Slurry Tests

The Contractor shall perform tests on the biopolymer slurry both before and after placing in the trench and shall consist of Marsh Funnel tests and pH determinations. Slurry samples from the trench shall be taken at approximate depth of 5 feet below ground surface. At least two sets of tests shall be taken each day of operation.

Marsh Funnel results shall be at least 50 seconds. pH shall be between 9 and 10.5.

C. Backfill Tests

The Contractor shall provide results of gradation tests of the free-draining gravel taken from at least three different truck loads. A copy of the test results shall be furnished to the Trust's Engineer as soon as available. Any material placed that does not meet the material requirements shall be replaced at no additional cost to the Trust. No gradation will be required on imported silty clay fill; however, analytical testing will be required.

D. Equipment

The Contractor shall furnish and maintain without charge the following equipment to be used by qualified personnel, provided by the Contractor, who are trained to operate all equipment and who have a working knowledge of test procedures for slurry testing in accordance with applicable American Petroleum Institute (API) Standard Specifications:

One (1) marsh funnel set
One (1) slurry sampler
One (1) soil sampling tool and driving apparatus
One (1) pH kit

E. Trench Bottom and Backfill Profiles.

1. The Contractor shall furnish a profile of each trench bottom as well as the backfill slopes at the beginning of each day.

2. Depth measurements for the trench bottom shall be taken every 10 feet horizontally.

3. Depth measurements for the backfill placement shall be taken every 10 feet horizontally.

4. Records of any corrective actions taken shall be furnished to the Trust's Engineer.

F. *Pipe Elevation Profile*

1. The Contractor shall furnish a profile of the horizontal SVE screen, including elevations, for approval prior to backfilling above the SVE screen at each trench.

2. The Contractor shall also furnish elevation measurements of the PRGS conveyance piping at the end of each trench segment and the manhole connection.

3.11 PERFORMANCE TESTING/ACCEPTANCE

A pumping test shall be performed at each trench segment upon completion of development and flushing. The dewatering well (or temporary biopolymer development well) will be pumped at approximately 5 gallons/minute to dewater the trench. If the trench can not be dewatered at this pumping rate, then corrective measures will be required.

3.12 CORRECTIVE MEASURES

Corrective measure will depend on the conditions encountered and must be approved by the Trust prior to start of the work. If corrective measures are requested, the Contractor shall submit a work plan to the Trust for approval prior to implementing the actions.

END OF SECTION

SECTION 02268

MONITORING WELLS

SECTION 02268

MONITORING WELLS

PART 1 – GENERAL

1.01 SUMMARY

A. The work covered under this section is expected to be arranged by the Trust's Engineer. Costs for well installation, installation oversight, development, and sampling will be billed to the Trust separately.

B. The well installer shall possess all necessary licenses and shall obtain the appropriate permits needed to comply with the State of Indiana regulations.

1.02 SITE CONDITIONS

A. Drilling Location: Monitoring Wells S-4B and S-5 locations were provided in Attachment Z-1 and the Addendum to the Field Sampling Plan.

B. Subsurface conditions are shown in Drawing C-4.

1.03 DESCRIPTION OF WORK

A. Monitoring Wells S-4B and S-5 shall be constructed in the sand and gravel water-bearing unit. Construction details are provided below.

B. Access to the drilling locations may require the use of all-terrain or tracked vehicles. The well installation Subcontractor is responsible for providing all equipment necessary to gain access to the drilling locations.

C. Power generation is the responsibility of the well installation Subcontractor.

D. There is no water source on site. The Subcontractor is responsible for providing all equipment necessary for temporary storage and transportation of water.

E. Well installation Subcontractor is responsible for providing personal protective equipment (PPE). Drilling is anticipated to be completed in Level D protection. Provisions are to be made to upgrade to higher levels of protection as determined by the Health and Safety Site Officer.

1.04 SUBMITTALS

Well construction log, including well material information, upon completion of well installation.

PART 2 – PRODUCTS

2.01 MATERIALS

A. Protective casing: 4-inch (nominal) diameter Schedule 40 steel.

B. Temporary casing: 10-inch diameter steel.

C. Polyvinyl chloride (PVC), Schedule 40, flush-threaded well pipe, 2-inch diameter.

D. PVC, Schedule 40 slotted well screen 0.01 slot, 2-inch diameter.

E. Sandpack: Filter sandpack #4 or equivalent (for sand zone wells).

F. Bentonite: Bentonite pellets will be used for the annular seal. Approved proportions of powdered chip bentonite will be used in temporary casing seals, drilling mud, and annular grout.

G. Grout: Annular grout will consist of Portland cement, bentonite, and clean potable water. *(Note: Water is not available at the site.)*

PART 3 – EXECUTION

3.01 GENERAL

A. Decontamination

1. Decontamination of equipment will be accomplished using high pressure, hot water, or steam at the designated on-site decontamination pad.

2. The rig and associated equipment is to be decontaminated upon arrival and departure from the Site. Decontamination of downhole drilling equipment will take place between borehole locations.

3. The Contractor is responsible for ensuring the decontamination and integrity of well materials.

B. Sampling

1. Confirmatory split-spoon samples will be taken according to ASTM D-1586.
2. Sampling frequency will be continuous throughout each borehole location.

C. Waste Handling

1. Containerization and handling of solid and liquid wastes produced during drilling and development activities are the responsibility of the Subcontractor. Wastes shall be left on-site to be managed by the General Contractor.

2. Solid wastes shall be placed in a roll-off box, the *ex situ* soil treatment cell or in Department of Transportation (DOT) 17H 55-gallon drums and stored in the Support Zone as directed by the Trust's Engineer.

3. Liquid wastes shall be placed in DOT 17H 55-gallon drums and/or transferred by the Contractor to the wastewater storage tank(s) in the Support Zone.

D. Development

1. The monitoring wells are to be developed using the surge and pump method.

2. Development of each piezometer will continue until ground water temperature, pH, and specific conductance values equilibrate.

3. In the event that monitoring wells are bailed or pumped dry before all the development parameters (listed above) equilibrate, the piezometers will be permitted to recover to at least 25 percent of the original static water and pumped dry again before development resumes. Development will be terminated after pH and specific conductivity equilibrate.

3.02 SAND ZONE WELL INSTALLATION

A. Drilling Method

1. Sand (water-bearing zone) monitoring wells shall be drilled using the Water Rotary Drilling method. A 12-inch diameter wing bit or blade bit circulating water shall advance the borehole. Soil samples shall be collected through the drill stem and bit to locate the till/sand interface depth. A temporary 10-inch ID steel casing shall be set near the base of the till and sealed with a bentonite slurry to prevent shallow till zone cross contamination of the sand zone. After the bentonite slurry is set, a 7-7/8-diameter blade or wing bit will be utilized to advance the borehole through the casing into the sand zone.

B. Installation

1. Each sand and gravel well is to be installed through the steel surface casing in the open borehole. Riser pipe and screens shall be decontaminated prior to installation unless environmentally sealed prior to installation. Measures are to be taken to avoid a cross contamination of the screen and riser from contact

with the ground, the rig, and other objects. Clean gloves are to be worn when handling the screen and riser.

2. After installation of the PVC materials, gravel pack and bentonite seal, the remaining annular space will be backfilled with a bentonite cement mixture via tremie pipe.

C. Annular Material Installation

1. Annular materials (sandpack and grout) are to be emplaced using a tremie pipe. The cement-bentonite grout slurry is to be mixed at the surface prior to emplacement.

2. The bentonite pellet seal will be installed by dropping the pellets through the casing to the top of the sandpack. The casing will be pulled from the borehole incrementally until the bentonite pellet seal is a minimum of 2 feet above the sand/lower till interface. The bentonite pellet seal will be permitted to hydrate for a minimum of 1 hour before backfilling the remaining annular space with cement bentonite grout.

D. Protective Casing Installation

The 4-inch protective steel casing is to be extended to the ground surface and shall be finished with a flush-mounted cover and concrete pad.

END OF SECTION

SECTION 02280

GEOTEXTILES

SECTION 02280

GEOTEXTILES

PART 1 – GENERAL

1.01 DESCRIPTION

A. This section includes the procurement, transportation, storage, handling, seaming, and installation of the geotextile for: (1) protection of the geomembrane liner for the augmented Soil Vapor Extraction (SVE) trenches, (2) protection of the geomembrane for the *ex situ* SVE treatment cell, and (3) leak detection spacing between the T5 tank primary and secondary liners.

1.02 RELATED SECTIONS

A. Section 01300 – Submittals

B. Section 02210 – Augmented SVE Trench

C. Section 02750 – Erosion Control Revetment

D. Section 13050 –Wastewater Storage and Transfer System

E. Section 13100 – *Ex Situ* SVE Treatment Cell

1.03 REFERENCES

A. ASTM D3776 Test Methods for Mass Per Unit Area (Weight of Woven Fabric).

B. ASTM D3786 Test Method for Hydraulic Bursting Strength of Knitted Goods and Non-Woven Fabric (Diaphragm Burst Strength Tester Method).

C. ASTM D4355 Test Method for Deterioration of Geotextiles from Exposure to Ultraviolet Light and Water (Xenon-Arc Type Apparatus).

D. ASTM D4533 Test Method for Trapezoid Tearing Strength of Geotextiles.

E. ASTM D4632 Test Method for Breaking Load and Elongation of Geotextiles (Grab Method).

1.04 SUBMITTALS

A. The Contractor shall submit the following:

1. Product Data

    a. Manufacturer's descriptive literature and specifications covering the product specified, including installation information.

2. Certificates of Conformance

    a. Manufacturer's certification that the geotextile was manufactured in accordance with specified reference standards.

PART 2 – PRODUCTS

2.01 MATERIALS

A. The geotextile shall be a nonwoven geotextile comprised of polypropylene filaments which are formed into a stable network which meets or exceeds the following minimum average role value (MARV) properties:

Fabric Properties	Test Method	MARV
Weight, oz/yd^2 (min.)	ASTM D-3776	7.7
Grab Tensile Strength, lbs.	ASTM D-4632	200
Grab Tensile Elongation, %	ASTM D-4632	60
Trapezoidal Tear Strength, lbs.	ASTM D-4533	85
Mullen Burst Strength, psi	ASTM D-3786	250
Ultraviolet Stability, %	ASTM D-4355 (Xenon Arc) 500 hrs. exposure	70

PART 3 – EXECUTION – Not Applicable

END OF SECTION

SECTION 02730

VAULTS AND MANHOLES

SECTION 02730

VAULTS AND MANHOLES

PART 1 – GENERAL

1.01 DESCRIPTION

A The Contractor shall furnish all materials, labor and equipment necessary to install the precast concrete vaults and manholes. Section includes precast manholes and utility vaults, frames, covers, hatches, anchorage, and all accessories.

B. The SVE screen – PRGS conveyance pipe connection shall be housed in manholes located as shown on the Design Drawings. The manholes shall also house connections between the PRGS conveyance piping segments. An additional manhole will be used for the connection between the two legs of PRGS conveyance piping.

1.02 RELATED WORK NOT INCLUDED

A. Section 02200 – Excavation, Backfill, and Compaction

1.03 SUBMITTALS

A. Product data: Provide manhole configuration and dimensions at least 10 days prior to the Pre-Construction Conference.

B. Shop Drawings: Indicate manhole locations, elevations, dimensions, penetrations, and installation information as part of the as-built/completion report.

1.04 REFERENCE STANDARDS

A.	ASTM A48	Gray Iron Castings
B.	ASTM Al 85	Welded Deformed Steel Wire Fabric
C.	ASTM C 185	Standard Test Method for Air Content of Hydraulic Cement Mortar
D.	ASTM C 478	Standard Specification for Precast Reinforced Concrete Manhole Sections

E. ASTM A615 Deformed and Plain-Steel Bars for Concrete Reinforcement

F. ASTM C923 Resilient Connectors for Reinforced Concrete Manhole Structures and Pipes

1.05 QUALIFICATIONS

A. Manufacturer: Company specializing in manufacturing products specified in this section with minimum of 3 years of experience.

1.06 TRANSPORTATION, DELIVERY, AND HANDLING

A. Transport, store, and handle precast sections in a manner to prevent spills, cracks, and other damage to the units.

B. Contractor shall make repairs and patches to pre-cast units to bring in to compliance.

PART 2 – PRODUCTS

2.01 MATERIALS

A. Manhole and Vault Sections: All units shall be reinforced to conform to ASTM A615. Precast vaults and manholes shall be constructed in accordance with ASTM C478 and connections in accordance with ASTM C923. Sleeves for pipe openings to be factor cast for pipes 6-inches and larger; smaller diameter pipe openings and sleeves can be prepared in the field.

B. Base sections shall have reinforced flat bottoms protruding a minimum of 6 inches beyond the outside face of the riser section. The bases shall be minimum 6 inches thick for risers up to and including 48" diameter

2.02 PRECAST MANHOLE DESIGN CRITERIA

A. Standards: Building Code Requirements for Reinforced Concrete (ACI318) and AASHTO Standard Specifications for Highway and Bridges.

B. Materials

1. Concrete: 4,000 psi @ 28 days minimum
   3,500 psi @ time of delivery

2. Reinforcing bar: ASTM A615, Grade 60
   Welded wire fabric: ASTM A 185

C. Design Loads

High Water Table: approximately 4 to 6 feet below ground surface
Coefficient of lateral earth pressure for wall design: 0.45

2.03 ACCESSORIES

A. Lockable Metal Frame and Cover

Manhole frames and cover shall conform to ASTM A48 and be designed to carry AASHTO U20 live loads. No cover will be accepted that "rocks" in place after construction.

B. Steps

Minimum 0.5-inch diameter round steel encapsulated with co-polymer of polypropylene plastic. Steps shall be capable of withstanding the design loading requirements of ASTM C478 at a temperature of 0 °F with no structural failure.

1. Steps shall be cast into the walls of base, risers and conical top sections of manholes and sidewalls of vaults

2. Steps shall be secured with mortar or cast in place polypropylene inserts. Steps shall meet requirements of OSHA 1910.27 for fixed ladders

C. Sealing Compound

Two rings of butyl rubber joint sealant shall be set between the bottom of frame and top of concrete section or grade rings. Joint sealant shall also be set between precast sections.

D. Grade Rings

Concrete masonry units conforming to ASTM C478; hold down bolt holes matching the manhole frame. No split rings permitted.

E. Pipe Opening Seals

Factory cast or field installed resilient gasket-type seal that conforms to ASTM C923 and sized to match pipe openings required on the Design Drawings. Flexible rubber connectors shall be: Kor-N-Seal, Lock Joint Flexible Manhole Sleeve, or approved equal.

F. Bitumastic Coating

Two (2) coats of asphalt-based coating factory applied to all precast sections. Coating shall be Karnak #83 AF Fibrated Damproofing, Koppers 300 M Epoxy, or Pennsbury32-B-4 Epoxy.

2.04 CONFIGURATION

A. Concentric risers and base with eccentric cone style top section for cylindrical manholes. Joints between sections shall be lipped male/female joints; manhole sleeves to receive process pipe. Design depths shall be as indicated on the Drawings.

B. Manholes shall be cylindrical or rectangular, located as indicated on the drawings.

1. Cylindrical manholes to have minimum clear inside diameter of 48 inches with a 30-inch man way opening. Cover shall be cast iron designed to withstand H20 loads and sized to cover the opening.

2. Rectangular vaults shall have a metal cover designed to withstand H20 loads and sized to cover the opening.

PART 3 – EXECUTION

3.01 INSTALLATION – GENERAL

A. Verify that items provided by manufacturer are properly sized and located.

B. Excavations shall begin 6 inches from all sides of the base and extend to a maximum of 1 foot from all sides of the top.

C. Scarify and re-compact subgrade. Remove any unsuitable soils (peat, organics, fat clays, or silts). Install bases on a minimum 6-inch layer of compacted crushed stone.

D. Place base pad on prepared base material. Make sure base section is level.

E. Place manhole/vault sections, plumb, level, and trim to correct elevations. Anchor riser to the base pad.

F. Install sealing compound into annular spaces to completely fill any voids in wall openings or joints and render installation watertight.

G. Backfill and compact area around the manhole with sand bedding up to the existing ground surface.

H. Contractor is to set cover frames and covers level on grade rings if necessary to correct elevations as determined by finished grade. Set grade rings in waterproof mortar immediately before installing manhole frame.

3.02 VAULT AND MANHOLE LEAKAGE TESTING

A. Inspect each vault and manhole after all pipes are connected to identify any infiltration.

B. Repair and retest: Determine source of leaks in vaults/manholes. Repair or replace defective material.

END OF SECTION

SECTION 13050

WASTEWATER STORAGE AND TRANSFER SYSTEM

SECTION 13050

WASTEWATER STORAGE AND TRANSFER SYSTEM

PART 1 – GENERAL

1.01 DESCRIPTION OF WORK

A. The Contractor shall provide all labor, equipment, and materials to upgrade and operate the existing on-site storage and transfer system for wastewaters generated during construction and operation of the Augmented Soil Vapor Extraction trench system. The wastewater storage system currently includes two modular tanks for storage of both raw and treated wastewaters. The transfer system includes pumps, piping, and controls, as needed, to convey treated waters from the storage tanks to either an on-site channel for discharge, to a tanker for hauling to an off-site disposal facility, or to the raw water storage tanks for retreatment.

The existing wastewater storage and transfer system is currently operational and is configured to treat wastewater from the neighboring Third Site. The wastewater treatment system may be operated by both ECC and Third Site, provide separate waste streams are maintained. Simultaneous operation by both ECC and Third Site can occur through alternating batch treatment. An additional raw wastewater tank (T5) and associated piping will be required to maintain separate waste streams. The upgrades to the existing wastewater treatment system are presented below.

The process flow and instrumentation diagram (P&ID) for the wastewater storage and transfer system is shown on the Drawings.

1.02 SUBMITTALS

The Contractor shall provide the following submittals to the Trust's Engineer at least 10 days prior to the Pre-Construction Work Conference.

A. Wastewater Storage and Transfer System Installation Plan, including product details regarding the following:

1. Tank wall, frame supports
2. Tank liner and cover materials
3. Piping and pipe fittings
4. Access stairways
5. Tank vent (T-5) system
6. Leak detection sump

B. Shop Drawings

1. Manufacturer's published detailed drawings, modified to suit design conditions, as necessary, and Contractor's prepared drawings, as applicable, depicting inlet piping connections and supports, bottom drains, leak detection drains and sumps, transfer pumping systems, and spill control. To be submitted as part of the wastewater storage and transfer plan.

2. Contractor's plan for construction, dewatering, and ASVE system wastewaters.

C. Installation Instructions

Manufacturer's installation instructions for the wastewater storage tank system.

PART 2 – PRODUCTS

2.01 WASTEWATER STORAGE TANK

A. The Contractor shall provide one steel storage tank, 43-foot diameter by 15-foot high with a total volume of 150,000 gallons capacity as measured at 6-inch freeboard. The tank shall be ModuTank HiStor Storage tank, or equivalent, as manufactured by:

ModuTank, Inc.
Long Island City, NY 11101
(800) 245-6964

ModuTank shall provide the tank steel and hardware, the tank liners, cover, and geotextiles, and the bottom drain and leak detection fitting assemblies. All other site preparation materials and piping shall be provided by the Contractor.

B. Tank shall be free-standing and self-supporting. The tank shall be able to be installed on a level, compacted earth site. The tank wall shall be placed on an existing perimeter ring footer consisting of IDOH No. 5 coarse aggregate approximately 48 inches wide and 12 inches deep.

C. The tank shall consist of modular steel components and be shipped as a knock-Down (KD) unit and be assembled using simple hand tools.

D. The tank walls shall be set on steel footing plates placed directly on the ring footer. The footing plates shall be 24 by 30 by 1/16-inch CORTEN steel per ASTM A606. The plates shall be anchored on the outside of the tank wall with a concrete block or equivalent dead weight which provides a minimum 300-pound load per plate.

E. Tank walls shall be 12-gauge LFQ G-90 Mill Galvanized Steel panels per ASTM A569, 5 by 10 feet dimension. Wall support girths shall be 3 inches by 3 inches by 3/16-inch structural steel angle, hot dip galvanized after fabrication. The girths shall be ASTM A36 structural steel angles. The girths shall be placed horizontally to connect the wall panels and will consist of one bottom girth, two middle girths, and a top girth.

F. Wall and support frames shall be bolted together using bolts, nuts, and washers to be supplied by the tank vendor.

2.02 TANK LINER SYSTEM

A. The storage tank shall have a double-liner construction for dual containment of liquids. The liner shall be shop-fabricated in one piece to fit the tank. The liners shall be as follows:

1.	Primary (top) Liner:	30 mil reinforced XR-5, or equivalent
2.	Secondary (bottom) Liner:	30 mil reinforced XR-5, or equivalent

B. A leak detection geotextile shall be placed continuously between the primary and secondary liners. The secondary liner shall have a continuous geotextile underlayment on the subbase. The geotextiles are described in Section 02280 – Geotextiles.

2.03 PIPING

A. Bottom Drain. The T-5 tank shall have a 4-inch diameter Schedule 80 PVC bottom drain. The bottom drain shall be installed in accordance with the manufacturer's recommendation and be placed in a 3-foot deep trench as shown on the Drawings. The bottom drain shall have a 4-inch diameter stickup screened to minimize solids and debris entry into the drain.

B. Leak Detection System. The T-5 tank shall have a 4-inch diameter Schedule 80 PVC leak detection drain that connects to a vertical 12-inch diameter Schedule 80 PVC sump. The leak detection drain shall be installed in accordance with the manufacturer's recommendations. The drain pipes shall be placed in a 3-foot deep trench and be sloped at 1 percent to the sump. The sump shall stickup 6 inches above the tank wall and shall be capped. The sump shall be located as shown on the Drawings.

C. Inlet Pipe. All inlet pipes into the T-5 tank shall be over the top of the tank wall.

D. The tank shall be fitted with a system of 2-inch, Schedule 80 aeration lines. These lines will be placed at the base of tank T-5 and connected to new supply equipment. Diffuser heads will be added at five locations.

2.06 TANK T-5 COVER AND VENT TREATMENT

A. Storage Tank T-5 will be equipped with a floating cover and vent treatment system to control VOC vapor emissions from the stored wastewater. The tank cover shall be constructed as indicated on the Drawings and shall include the following components:

1. Floating cover fabricated of 30 mil reinforced XR-5, or equivalent, incorporating floatation logs, ballast pipe, protective liner bumper, and rope retention grid.

2. Vent assembly consisting of two (2) 4-inch diameter vents, flexible hose, and over-the-wall piping.

The tank cover and vent assembly shall be as manufactured by ModuTank, Inc., or equivalent, and shall be compatible for construction and operation with the wastewater storage tank.

B. Tank vent T-5 discharge shall be conveyed to an activated carbon treatment system. Two 55-gallon VentSorb canisters connected in series, as manufactured by Calgon Carbon Corporation, Pittsburgh, Pennsylvania, or equivalent, shall be used. The canisters shall be situated along the outside wall of Tank T-5 in a location approved by the Engineer. A standby clean canister shall be kept on-site for replacement, as needed.

The vent treatment system shall include a condensate drain and storage tank (minimum 55-gallon) upflow of the carbon canisters to remove moisture. A sample valve shall be located between the canisters to allow routine vapor testing for VOC breakthrough.

PART 3 – EXECUTION

3.01 OPERATIONS

A. The wastewater storage and transfer systems shall be constructed and all quality control inspections shall be completed prior to starting construction dewatering.

B. Maximum operating water levels in the tank shall be at 12 inches below the top of the sidewall rail. No waters shall be transferred into a tank that has reached its maximum water level.

Waters encountered during construction at a volume in excess of that which can be transferred into the T-5 tanks shall be temporarily stored by the Contractor by a means approved by the Trust's Engineer.

C. Solids Removal and Handling

1. The Contractor shall be responsible for removal and containerization of all solids collected in the storage tanks during the duration of remediation activities. Settled solids shall be removed if the settled volume exceeds 15 percent of the capacity of the tank.

2. All solids and residues collected during cleaning of the storage tanks shall be analyzed by the Contractor and disposed off site if results exceed the Site-Specific Exposure Concentrations.

D. Vent Treatment System at Tank T-5

The carbon canister vent treatment system shall be operated in the lead/lag mode. The lead canister discharge shall be monitored on a minimum weekly basis during operation of the wastewater treatment system. A photoionization detection (PID) shall be used to measure total VOCs emitting from the sample valve between the canisters. Breakthrough shall be designated as a PID VOC measurement of 10 ppm.

Upon determination of VOC breakthrough, the lead canister shall be replaced with the lag canister and a clean canister (standby) shall be installed as the lag canister.

3.04 DEMOBILIZATION

A. The Contractor shall remove from the site T-5 wastewater storage tank and associated materials, with the exception of the ring footer and below-grade piping at the completion of the Phase I Monitoring.

END OF SECTION

SECTION 13100

EX SITU SOIL VAPOR EXTRACTION CELL

SECTION 13100

EX SITU SOIL VAPOR EXTRACTION CELL

PART 1 – GENERAL

1.01 This specification provides requirements for the ex-situ SVE cell. The Contractor shall furnish all labor, materials, and equipment required to construct the cell, transport the soils and install the connections to the existing SVE system as shown on the Design Drawings and specified herein. Note that *ex situ* soil treatment will only be required if the conditions discussed in Section 2.8.1 of the Design Report are present. A smaller cell than shown on the Design Drawing may be appropriate based on actual soil volumes to be treated.

1.02 RELATED SECTIONS

A. Section 02110 – General Site Preparation

B. Section 02210 – Augmented SVE Trench Construction

1.03 *EX SITU* SVE SOIL TREATMENT CELL DESCRIPTION

A. The *ex situ* SVE soil treatment cell will encapsulate contaminated soils excavated from the SVE trenches and systematically move air through the soils to enhance volatilization and removal of volatile organics. Air movement through the soil shall be facilitated by a network of screened PVC piping connected to the existing SVE vacuum system. The *ex situ* SVE process shall involve the continuous extraction of organics-laden air and treatment of the air by a vapor treatment unit to remove organics.

The existing SVE system at the ECC Site shall be used to move the air and to collect water vapor if present. Any water collected will be contained by the system water separation tank and later transferred to the ECC wastewater treatment facility.

The *ex situ* SVE soil treatment may be conducted concurrently with SVE of the augmented SVE trench segments. The treatment shall continue until vapor analysis indicates compliance with the approved ECC Site Soil Vapor Standards.

Ex situ SVE soil treatment cell shall include the following major components:

1. Geomembrane above and below waste soils.

2. A 4-inch diameter lateral piping that is slotted within the cell and solid along alignment to treatment building.

3. A 2-inch diameter slotted ambient air inlet piping,

4. One vapor monitoring point.

1.05 SUBMITTALS

A. *Ex Situ* SVE Soil Treatment Configuration Plan

1. To be submitted after trench excavation soil testing is complete and the volume of soil to be treated is defined.

2. Shall include final dimensions and location of the cell and piping network, along with excavated soil analysis laboratory data.

3. Shall include details regarding connection to the existing SVE manifold in the treatment building and covering of the cell when treatment is complete.

4. Shall include specifications for the geomembrane.

PART 2 – GENERAL REQUIREMENTS

A. The *ex situ* SVE soil treatment cell system shall be constructed to allow air flow to affect all areas of soil within the cell.

B. The Contractor shall designate a qualified system operator and backup operator that shall be approved by the Trust.

C. Vapor monitoring data will be collected as required by the Trust and/or USEPA to assess the concentrations of contaminants of concern in the treatment cell soils.

1. If the vapor levels meet the approved Soil Vapor Standards, the soils in the treatment cell will be left in place, upon approval by the USEPA, in consultation with IDEM.

2. If the vapor levels do not meet the Soil Vapor Standards, the Contractor shall continue to operate the system until the soil meets the criteria.

PART 3 – EXECUTION

3.01 CELL CONSTRUCTION

A. The *ex situ* SVE soil treatment cell shall be installed with the configuration as defined in the approved Contractor submittal. Excavation of the existing cap soil will not be permitted; however, clean fill soils may be used to establish a level grade, as needed. A geotextile layer may be placed below the base of the cell for protection of the lower geomembrane, if desired.

B. Contractor shall place the geomembrane and soils to be treated so that the geomembrane surrounds the soils below, above and on the sides. Penetrations for the air inlet pipe(s), vapor monitoring point and SVE piping shall be patched with a geomembrane boot firmly attached to the PVC piping.

C. Care must be taken to avoid breakage of the PVC-slotted piping during soil placement. Any repairs shall be in responsibility of the Contractor.

D. Contractor shall place the slotted 4-inch diameter schedule 40 PVC pipe on *a minimum of 1 foot of soil at the base of the cell.*

E. Solid 3-inch diameter PVC pipe shall be attached to the respective ends of the 4-inch diameter SVE slotted pipe and extend to the treatment building SVE manifold. The penetration of the treatment building wall shall be patched and the opening insulated.

F. The 2-inch diameter air inlet pipes shall be fitted with an adjustable ball valve at the end outside the cell to regulate the volume of outside air allowed into the cell. The Contractor shall adjust the valves, as appropriate, for efficient soil treatment.

3.02 VAPOR MONITORING POINT INSTALLATION

A. Contractor shall install at least one vapor monitoring point into the main section of soils undergoing treatment.

B. Use pre-packed well screen Schedule 40 PVC, minimum 1-inch I.D., and 2.5-inch O.D. sand pack, minimum 5-foot screen length.

C. Install during soil placement, with cap and vacuum gage at riser top.

D. Patch geomembrane penetration with a geomembrane boot secured to the upper geomembrane layer and the monitoring point. Check for leaks with SVE system in operation and repair, if needed.

3.03 GEOMEMBRANE

A. Contractor shall overlap and weld or tape seams according to geomembrane manufacturer specifications. Seams shall be checked during SVE operation and repaired if leaks are noted.

B. Contractor shall limit traffic on the geomembrane and prevent soil accumulation that may result in vegetation growth with roots that could damage the geomembrane.

C. Where traffic is expected on the geomembrane, such as the path to access the vapor monitoring point, Contractor shall place a 3-foot wide second layer of the geomembrane to protect the primary layer.

3.04 OPERATION/TREATMENT

A. Contractor shall connect the *ex situ* SVE soil treatment cell SVE piping to the existing ECC treatment system and furnish vacuum capable of providing air flow within the cell.

B. Contractor shall perform initial start up with oversight provided by the Trust's Engineer. Startup shall commence after all components of the system have been installed and reviewed by the Trust's Engineer.

C. Contractor shall operate the SVE system on the treatment cell as appropriate for completing treatment efficiently and within approximately 12 months.

D. Contractor shall provide monthly status reports to Trust's Engineer providing periods of operation, flow rates, vacuum measurements, and monitoring data. Contractor shall make adjustments to system operation at the request of the Trust.

E. Contractor shall operate the SVE system continuously, 24 hours a day, 7 days a week, unless total shutdown occurs for cleaning, necessary adjustments or maintenance. Contractor shall be responsible for performing all manufacturer recommended maintenance activities during operation of the system.

F. Contractor shall remove ground water and condensate from the SVE piping, as necessary.

G. Contractor shall designate a qualified system operator and backup operator that shall be approved by the Trust. The operator and/or backup operator shall be available by direct communication or remote pager 24 hours a day in the event of a system shutdown or emergency.

3.05 SITE RESTORATION

A. Contractor shall restore the treatment cell area as soon as practical after the Soil Vapor Standards have been achieved and completion of the *ex situ* SVE program has been approved by USEPA, in consultation with IDEM. All above-grade piping shall be disconnected, removed and disposed off site. A minimum of 12 inches of clean soil cover shall be placed over the cell area and seeded. Excavation or grading of the existing clay cap will not be allowed.

END OF SECTION

SECTION 13110

WASTEWATER TREATMENT SYSTEM

SECTION 13110

WASTEWATER TREATMENT SYSTEM

PART 1 – GENERAL

1.01 DESCRIPTION OF WORK

A. This section covers the requirements for upgrading the existing on-site wastewater treatment system. The existing treatment system consists of filtration, air stripping, and activated carbon adsorption. It is intended primarily for the removal of organic contaminants and suspended solids. This system shall be used to treat wastewaters generated during the Construction and Active phases of the project, including during the excavation and operation of the Augmented Soil Vapor Extraction Trench System.

The existing system is currently operational and is configured to treat wastewater from the neighboring Third Site. The wastewater treatment system may be operated by both ECC and Third Site, provide separate waste streams are maintained. Simultaneous operation by both ECC and Third Site will occur through the use of the ECC wastewater storage and transfer system and alternating batch treatment. The upgrades to the existing system are necessary to allow for separate waste streams. The upgrades to the existing wastewater treatment system include those presented below. Other changes may be needed.

The process flow and instrumentation diagram (P&ID) in the Design Drawings includes the wastewater treatment system is shown.

1.02 RELATED SECTIONS

A. Section 13050 – Wastewater Storage and Transfer System

1.03 PERFORMANCE REQUIREMENTS

A. The two (2) liquid-phase granulated activated carbon (GAC) units shall contain 1,000 pounds of GAC and be capable of treating 50 gpm each, while operating in series. The carbon units shall be capable of treating the site wastewaters to achieve the IDEM effluent standards established for discharge on-site (Table 2-2 of the ASVES Design Report), when preceded by the air stripper. The liquid-phase GAC units shall have a maximum operating pressure no less than 85 psi.

B. The two (2) vapor-phase carbon treatment units shall be capable of treating 900 cfm in series. Construction shall be steel. The vapor-phase GAC units shall treat the stripper off gases to below IDEM limits.

C. The Contractor shall provide bubble diffusers aerators for storage Tank T-5. The aerators shall be submerged and shall supply at least 0.14 mg/L oxygen to the wastewater per mg/L of iron oxidized. The aerators shall be installed on the northern half of the tank bottom in a spoke and hub pattern.

1.04 SUBMITTALS

Wastewater Treatment system Upgrade Plan that includes manufacturer's catalog data for new system components and new system notifications.

PART 2 – PRODUCTS

2.01 WASTEWATER TREATMENT SYSTEM

A. The Contractor shall supply the following wastewater treatment system components:

1. Two (2) Carbonair Model GPC-48 vapor-phase GAC vessels or their approved performance equivalent.

2. Two (2) Carbonair Model PC-7 liquid-phase GAC vessels or their approved performance equivalent.

Both GAC systems shall be configured to operate in series so that one treatment unit will be the primary adsorber (lead) while the other treatment unit will act as a backup (lag). In both systems, the piping shall enable one unit to be isolated for disconnection and changeout while maintaining operation through the other.

B. Bubble Diffuser

1. The Contractor shall provide a bubble diffuser secured to the wall or floor of storage Tank T-5. The aerator shall be designed to maintain sufficient pressure on headers to eliminate clogging of diffusers. Oxygen transfer rate shall be at least 0.9 kg oxygen per kilowatt hour. A check valve shall be included in the over-the-wall piping from the air blower to the diffuser to avoid water flow to the blower. Power connections may need to be added for the blower for the diffuser.

PART 3 – EXECUTION

3.01 INSTALLATION

A. The Contractor shall be responsible for installing the systems within the Wastewater Treatment Building Area at the location shown on the Drawings. Pumps, piping,

and connection requirements for the wastewater storage system are provided in Section 13050. Process and instrumentation requirements are indicated on the Drawings. All instrumentation and manually-controlled appurtenances shall be installed in locations easily accessible by site personnel. New power connections may be required.

B. All piping and ductwork shall be clearly labeled with pipe markers clearly showing the following:

Pipe flow direction (with arrows);
Service/contents (liquid or vapor); and
Content name (minimum 3/4-inch all capital lettering (i.e., FEED WATER).

3.02 WASTE DISPOSAL

The Contractor shall be responsible for carbon acceptance testing and disposing and/or reactivation of spent carbon. Carbon shall be considered spent whenever breakthrough is detected from the lead treatment unit at the concentrations established during operations startup.

3.03 OPERATIONS

System startup and operations will be described in a Contractor Submittal for an Addendum to the existing Site Operations and Maintenance Plan.

END OF SECTION

SECTION 15050

PIPING – GENERAL PROVISIONS

SECTION 15050

PIPING – GENERAL PROVISIONS

PART 1 – GENERAL

1.01 WORK INCLUDED

A. The Contractor shall do all work required for the installation of pipe including excavation, furnishing and placing choked stone bedding, laying and jointing and testing of pipe, backfilling, and compacting.

B. The work shall also include storage in temporary spoil banks, all excess backfill, selection and placement of all suitable fill materials around the pipe, the disposal of surplus material at approved locations, and shall include all labor, tools, materials, and equipment necessary to install sanitary sewer piping.

C. The work shall also include furnishing, placing and removal of any trench support systems, the dewatering and pumping of all excavations, the protection of existing pipelines and structures, and the continuous cleanup of the site.

1.02 RELATED DOCUMENTS AND SECTIONS

A. Design Drawings and General Provisions of Contract

1.03 SYSTEM DESCRIPTION

A. Dimensions shown on Design Drawings are approximate only. Contractor shall verify all piping geometry in the field and shall be responsible for insuring proper alignment and fit of all piping consistent with the intent of the Design Drawings.

1.04 SUBMITTALS

A. The contractor shall submit a written testing plan prior to testing showing the AWWA testing procedures and documentation to be provided for hydrostatic testing of the installed PVC piping system.

PART 2 – PRODUCTS

2.01 CONTRACTORS' RESPONSIBILITY FOR MATERIAL

A. All pipes shall be carefully examined for defects, and no pipe known to be defective shall be laid. If any pipe is found to be broken or defective after being

laid, it shall be removed and replaced by sound pipe without any further payment than is included in the bid.

B. Joint surfaces shall be protected from damage, and shall be carefully examined before jointing. No damaged joints shall be used in the work.

C. Pipe shall be clean and ample precautions shall be taken to prevent entrance of dirt and debris into the pipe after laying. Exposed ends of all uncompleted lines shall be provided with plugs or covers at all times when pipe laying is not actually in progress.

D. The Contractor shall be solely responsible for the safe storage of all material furnished to or by him until it has been incorporated in the completed project and accepted by the Trust's Engineer.

E. Pipe and appurtenances shall be loaded and unloaded by lifting with hoists or skidding so as to avoid shock or damage. Under no circumstances shall such materials be dropped. Pipe handled on skidways shall not be skidded or rolled against other pipe.

PART 3 – EXECUTION

3.01 INSTALLATION - GENERAL REQUIREMENTS

A. All pipes shall be carefully laid to true alignment and grade with bell ends upgrade.

B. The trench backfill shall be carefully graded to the proper elevation, and the maximum practical solid bearing areas shall be provided throughout its entire length, prior to swinging the pipe into place. Trench safety precautions, such as trench boxes or benching, shall be used in accordance with OSHA guidelines, as appropriate.

C. In general, the pipe shall be laid on a minimum of 6 inches of pea gravel, sand or compacted stone. SVE trench piping will be placed directly on the free-draining backfill after the backfill is leveled.

D. Immediately after the pipe is brought to final position, it shall be thoroughly secured and properly bedded, and ample support shall be provided to prevent settlement or disturbances.

E. Pipe shall be protected during construction against possible flotation due to pouring of concrete or in case the trench becomes flooded prior to placing the backfill.

F. Jointing and laying shall be in strict accordance with recommendations of pipe manufacturer.

G. Jointing shall be done in strict conformance with manufacturer's recommendations. Pipe shall be handled with care to avoid damage to the lining and coating.

H. Cutting of pipe where required shall be done in a neat and workmanlike manner using an abrasive cutting wheel or other means which will produce a smooth end normal to the pipe axis with the cement lining undamaged. After cutting, the end of the pipe shall be dressed to remove all roughness and sharp corners and beveled in accordance with the manufacturer's instructions. Pipe ends shall be thoroughly cleaned prior to jointing and only manufacturer approved lubricants shall be used.

I. All pipe used shall be stamped with the ASTM code for PVC pipe ensuring factory inspection and quality control.

3.02 HYDROSTATIC TESTING

A. A post-installation hydrostatic test shall be performed on the installed system (minus the slotted SVE piping) in accordance with AWWA C605 (latest revision). The contractor will fabricate the system such that hydrostatic testing can be conducted on the PVC piping before connection to the SVE slotted pipe. Contractor shall be responsible for notification of the Trusts Engineer prior to the testing, making arrangements to supply potable water for testing and shall bear all costs which shall be considered as included and paid for under this item. Contractor shall be responsible for expelling all air from high points in the lines through the air release valves. Piping sections of slotted/ perforated piping will not be tested. Piping shall be tested prior to connection with the existing system unless otherwise approved by the Trust's Engineer. The Contractor shall furnish all labor, materials and equipment for performing these tests in the presence of the Trust's Engineer, including calibrated pressure gauges, test bulkheads, filling, draining, and air release connections and valves, calibrated drum and test pump.

B. Unless otherwise required to meet working conditions the PRGS carrier pipe and new dewatering lines (inside piping of double-contained pipe) shall be tested under a hydrostatic pressure as defined in AWWA C650 (latest revision). The duration of each pressure test shall be at least two hours.

Under the foregoing conditions, the allowable leakage shall be determined by the following formula:

$$L = [SD(P)^{1/2}] / 148{,}000$$

L = Allowable Leakage, Gallons per hour
S = Length of Pipe Tested, feet
D = Nominal Pipe Diameter, inches
P = Average Test Pressure, psi

C. After the 2-hour test period, the computed allowable leakage volume (measured by the test water meter) must be injected into the test section by means of the injection booster pump. After the allowable leakage volume is added into the test section, the contractor will record the final pressure reading. If the final pressure reading is less than the initial reading, the test section has failed the hydrostatic test. If the final pressure reading is equal too or greater than the initial pressure reading, the test section has passed the hydrostatic test. The Trust's Engineer must witness the pressure readings and leakage volume added.

D. Joints that leak shall be repaired and retested under the same conditions and under the same period of operation. If joints are found to be defective, they shall be replaced until the line passes the required test.

E. Any cracked or broken pipe, fittings, or valves shall be removed and replaced with sound pieces.

F. Wherever conditions will permit, in the opinion of the Trust's Engineer, the pipes shall be tested before the trench is backfilled. All joints shall be examined during the open trench test and all visible leaks entirely stopped.

G. The Contractor shall be required to provide all pipe taps, gauges, and corporation cocks, as well as any other materials and equipment necessary to expel all air and test the lines.

END OF SECTION

APPENDIX B

Ground Water Elevation Data Summary and Excavated Trench Soil Volumes

TABLE B-1

Sand and Gravel Ground Water Elevations Along Augmented SVE Trench Alignment
November 1998 to May 2002
Enviro-Chem Superfund Site
Zionsville, Indiana

Well Number	Associated Trench Segments	11/9/1998 Till Water Elevation (feet AMSL)	2/16/1999 Till Water Elevation (feet AMSL)	5/10/1999 Till Water Elevation (feet AMSL)	8/9/1999 Till Water Elevation (feet AMSL)	11/9/1999 Till Water Elevation (feet AMSL)	5/15/2000 Till Water Elevation (feet AMSL)	12/5/2000 Till Water Elevation (feet AMSL)	1/29/2001 Till Water Elevation (feet AMSL)	8/13/2001 Till Water Elevation (feet AMSL)	11/27/2001 Till Water Elevation (feet AMSL)	2/4/2002 Till Water Elevation (feet AMSL)	5/6/2002 Till Water Elevation (feet AMSL)	Minimum Till Elevation (feet AMSL)	Maximum Till Elevation (feet AMSL)	Median Till Elevation (feet AMSL)
S-1	1,2	878.98	881.44	881.11	879.27	878.25	879.07	881.02	881.45	880.48	881.23	882.01	882.49	878.25	882.49	881.1
PZ-1	3	878.68	881.86	880.56	878.89	878.00	NM	880.52	880.89	880.00	880.70	881.45	881.87	878.00	881.87	880.6
S-2	4	878.62	880.75	880.46	878.86	878.02	879.62	880.45	880.81	879.95	880.60	881.31	881.74	878.02	881.74	880.5
S-3	5, 6, 7	877.99	879.75	879.46	878.15	877.39	878.76	880.01	879.77	878.99	879.57	880.27	880.43	877.39	880.43	879.5

Notes:

AMSL = Above mean sea level
PVC = Polyvinyl chloride inner well casing
NM = No measurement

TABLE B-2

Estimated Excavated Trench Soil Volumes
Enviro-Chem Superfund Site
Zionsville, Indiana

SVE Trench Segment	Length (feet)	Average Depth (feet)	Trench Width[1] (feet)	Soil Volume (feet ^{3})	Soil Volume (yards ^{3})	Soil Mass[2] (tons)	Fluff Factor[3] (dimensionless)	Soil Stockpile Volume (feet ^{3})	Soil Stockpile Volume (yards ^{3})
1	135	11	2.5	3,713	138	186	1.25	4,641	172
2	165	11	2.5	4,538	168	227	1.25	5,672	210
3	115	10	2.5	2,875	106	144	1.25	3,594	133
4	145	8	2.5	2,900	107	145	1.25	3,625	134
5	110	10	2.5	2,750	102	138	1.25	3,438	127
6	205	15	2.5	7,688	285	384	1.25	9,609	356
7	110	10	2.5	2,750	102	138	1.25	3,438	127
Total Estimate				27,213	1008	1,361		34,016	1,260

Notes:
Note 1: Two-foot wide trench specified, however some widening to to side disturbance is expected
Note 2: Assumed soil mass conversion: 1.35 tons per cubic yard.
Note 3: Fluff factor reference: RACER 2001.

APPENDIX C

Addendum to April 28, 1977 Field Sampling Plan
Attachment Z-1 Remedy

Addendum to April 28, 1997 Field Sampling Plan
Attachment Z-1 Remedy
Enviro-Chem Superfund Site
Zionsville, Indiana

Prepared for

Enviro-Chem Site Trust Fund

Submitted to

United States Environmental Protection Agency, Region 5
and
Indiana Department of Environmental Management

Submitted by

ENVIRON International Corporation
Deerfield, Illinois

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(continued)

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APPENDICES

I. INTRODUCTION/BACKGROUND

This Addendum to the April 28, 1997 Field Sampling Plan (FSP), referred to herein as the "Addendum to the FSP," describes the rationale and procedures for sampling and other field investigations conducted in association with the Attachment Z-1 Remedy at the Enviro-Chem Superfund Site ("ECC" or the "Site"), located in Zionsville, Indiana. This Addendum to the FSP includes relevant field sampling procedures contained in the existing FSP for the site (Field Sampling Plan, Revision 4, dated April 28, 1997) and provides additional field sampling details pertaining to the augmented soil vapor extraction (SVE) system. Portions of the April 28, 1997 FSP are appended to this Addendum to the FSP where relevant.

The primary activities described in the Addendum to the FSP include: (1) subsurface water monitoring and water level measurement; (2) surface water monitoring; (3) soil vapor sampling; (4) soil sampling for waste characterization; (5) sampling of biopolymer slurry after addition of enzyme; (6) monitoring of Permeable Reactive Gate System (PRGS) effluent; and (7) wastewater discharge monitoring. This Addendum to the FSP has been included as Appendix C to the *Design Report for Augmentation of SVE System* (the "Design Report").

The primary objective of the augmentation of the SVE system is to treat subsurface water and soil contamination in the vicinity of the augmented SVE trench system and prevent off-site migration of contaminated subsurface water to Unnamed Ditch. The existing SVE system will be augmented by additional SVE trenches to be installed generally along the alignment of a ground water collection trench previously required as Additional Work in Revised Exhibit A to the Consent Decree. The augmented SVE trenches will be connected to the existing SVE system and will be operated using all of the basic operations of the existing SVE system. In order to provide additional protection to Unnamed Ditch, the Attachment Z-1 Remedy also includes a perimeter thin barrier curtain wall (TBCW), which was constructed in May 2006, and a permeable reactive gate system (PRGS).

After construction pf the augmented SVE trenches and the PRGS is complete, there will be several distinct phases of the Attachment Z-1 Remedy. The activities will be different for each period. The periods and the associated activities are as follows:

- Active Phase: This is defined as the period of operation of the augmented SVE trench system.

- Phase I Monitoring: This is defined as the 1-year period beginning when the Soil Vapor Standards have been achieved in the augmented SVE trenches. At the completion of the Phase I Monitoring period, Phase II Long-Term Monitoring will begin at the Site.

- Phase II Long-Term Monitoring: This is defined as the period following the completion of Phase I Monitoring. Phase II Long Term Monitoring is divided into Phase II(a) and Phase II(b), as described in the Design Report.

II. FIELD SAMPLING PROGRAM

The sampling and analysis schedule for the Attachment Z-1 Remedy is described in Table C-1. The sampling locations and analyses are described below. The sampling equipment and procedures are discussed in Section III. All sampling will be performed by the Trust's Engineer with the exception of the soil vapor sampling discussed in Section II.C below, which will be performed by the Contractor/Operator.

A. Subsurface Water Monitoring

Subsurface water samples will be collected from the PRGS, augmented SVE trenches, and sand and gravel monitoring wells as follows:

Combined Trench Segment Water Sample

A combined trench water sample may be taken from the PRGS collection manhole if, after completion of SVE, water accumulates in the augmented SVE trench segments. This sample will be collected using a dedicated bailer, which will be lowered into the PRGS collection manhole, after the connection valves to the conveyance piping from the east and west sides of the Site are opened and water accumulates in the sump. If no water is present within the PRGS collection manhole at the time of the sampling event (end of Active Phase), the PRGS combined water sampling will be considered complete.

Augmented SVE Trenches

Subsurface water samples from the individual augmented SVE trench segments will be collected from the vertical dewatering well installed within each augmented SVE trench segment. The locations of the trench dewatering wells are shown on Figure C-1. If insufficient water is present, the sampling for that trench segment will be considered complete for that event. Sampling of the trenches, if water is present, is scheduled for the Active Phase, the Phase I Monitoring period, and the 2-year Phase II(a) Long-Term Monitoring period. Active Phase, Phase I Monitoring and Phase II(a) samples shall be analyzed for the volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) with Acceptable Stream Concentrations (Table 2-1).

Sand and Gravel Monitoring Wells

A subsurface water sample will be collected from sand and gravel monitoring wells S-1, S-4B and S-5 during the Active Phase, Phase I, and Phase II(a) monitoring periods. The locations of sand and gravel monitoring wells S-1, S-4B and S-5 are shown on Figure C-1.

The samples will be analyzed for VOCs and SVOCs with Acceptable Stream Concentrations (Table 2-1).

B. Surface Water Monitoring

Surface water samples will be collected from Unnamed Ditch at three locations, designated SW-1, NSL-1 and SW-2, at the east boundary of the Site. The surface water sampling locations are shown on Figure C-1. The surface water samples will be analyzed for VOCs and SVOCs with Acceptable Stream Concentrations (Table 2-1).

C. Soil Vapor Sampling

During the Active Phase, pre-treatment vapor from each individual SVE trench will be collected periodically and analyzed for the VOCs and SVOCs with Soil Vapor Standards (Table 2-2) at on off-site laboratory according to the schedule in the Design Report. The trench vapor samples will be obtained from the existing sampling ports at the individual inlets at the existing SVE manifold at the Treatment Building or at the individual wellheads.

In addition, the combined vapor from all of the active trench segments will be analyzed for total organics using the in-line Series 8800 Continuous Analyzer as needed by the Contractor for system optimization. During the first week of operation of the SVE system, five vapor samples will be collected from the combined vapor stream for laboratory analysis concurrently with the in- line vapor measurements to establish a correlation between the in-line Continuous Analyzer and laboratory results. The laboratory samples for correlation will be analyzed for the Soil Vapor Standard list of parameters (Table 3-1).

Confirmation sampling after restart testing will be performed by the Trust's Engineer from the wellheads of the SVE trenches being tested. Summa canister samples will be analyzed for the VOCs and SVOCs with Soil Vapor Standards (Table 2-2).

If the *ex situ* SVE soil treatment cell discussed in Section 2.8 of the Design Report is used to treat excess soils excavated from the augmented SVE trenches, soil vapors extracted from the *ex situ* cell will be sampled for compliance purposes with restart spike testing as described for the augmented SVE system in the Design Report. *Ex Situ* cell samples will be taken from a manifold port in the Treatment Building where the vapors from the individual vacuum lines are combined. The soil treatment cell vapors will be analyzed for the VOCs and SVOCs with Soil Vapor Standards (Table 2-2).

D. Soil Waste Characterization Sampling

Soil samples will be collected and analyzed as part of excavated soil management activities. Initially, the stockpiles and upper soils will be scanned using a photoionization detector (11. eVlamp) as described in Section 2.8 of the Design Report. To characterize excess excavated soil,

soil samples will be collected for laboratory analysis from the stockpile of soil excavated from each augmented SVE trench at a frequency of one sample per SVE trench segment. All excavated soil samples will be collected as grab samples and will be analyzed for the VOCs, SVOCs, inorganic parameters and PCBs with Acceptable Stream Concentrations (Table 2-1) using the Synthetic Precipitate Leaching Procedure (SPLP), as well as the VOCs and SVOCs with Acceptable Soil Concentrations (Table 2-1) using standard (non-leachate) methods.

E. Trench Water Biopolymer Slurry Break Down Sampling

As discussed in Section 2.4.1 of the Design Report, following the addition of enzyme to the trench segments to break down the biopolymer slurry, one sample will be collected from each trench segment and analyzed for Biochemical Oxygen Demand (BOD), viscosity, and, if recommended by the Contractor, other indicator parameters.

F. PRGS Effluent Monitoring

As discussed in Section 5.2.2 of the Design Report, after Phase II(s) Long-Term Monitoring period are completed, one sample of treated effluent will be collected from the sample collection port at the outflow of the PRGS Treatment Vessel for laboratory analysis on an annual basis. Samples shall be analyzed for Acceptable Stream Concentration parameters (Table 2-1).

G. Wastewater Discharge Monitoring

As discussed in Section 2.8.3 of the Design Report, the water treatment system will be tested for compliance with wastewater discharge limits by conducting analysis of each batch of treated water stored in the 150,000-gallon treated wastewater tank prior to discharge. Sampling and laboratory analysis of the treated water will be conducted by the Contractor and the results submitted to Indiana Department of Environmental Management (IDEM) for approval to discharge. Effluent limits are provided in Appendix C-1.

H. Water Level Measurements

Water level measurements will be made from the TBCW piezometers quarterly during the Active Phase, the 1-year Phase I and Phase II(a) monitoring periods Also, water levels will be measured in the SVE trench dewatering wells during the Phase I and Phase II(a) Monitoring periods.

III. FIELD SAMPLING PROCEDURES

The overall sampling objective is to collect data of sufficient quality and quantity to achieve the highest level of confidence and, therefore, the lowest level of uncertainty in determining the completeness of the Attachment Z-1 Remedy activities. The sampling to be conducted during construction, augmented SVE system operation, and Phase I and Phase II Monitoring periods is designed to achieve this overall objective, as described in the following sections. A list of field equipment for each sampling program is provided in Table C-2. Field procedures are provided in Appendices C-2 through C-8 to this Addendum to the FSP.

CompuChem Laboratories of Cary, North Carolina and Air Toxics of Folsom, California will provide the analytical services.[1] Sample containers, sample preservation requirements and holding times are provided in Table C-3. Quality assurance measures are outlined in the Quality Assurance Project Plan (QAPP), included in Appendix E of the Design Report.

A. Subsurface Water Sampling Procedures

Combined Trench Segment Water Sample

The combined trench segment water sample will be collected from the PRGS collection manhole after all of the PRGS conveyance pipe valves are opened and sufficient water has accumulated in the PRGS sump. The combined PRGS water sample will be collected using a dedicated Teflon bailer, peristaltic pump or bladder pump (with dedicated tubing) and placed in laboratory containers. If sufficient water is not encountered, the sampling event will be considered complete.

Subsurface Water Monitoring - Augmented SVE Trenches

The subsurface water samples from the vertical dewatering wells installed within each augmented SVE trench segment will be collected using dedicated Teflon bailers. No purging of the wells will be conducted prior to sample collection. Field procedures for collection of water samples using dedicated bailers are contained in Appendix C-2. Procedures for field measurement of water parameters are also included in Appendix C-2.

[1] The biopolymer slurry analyses may be conducted at a different laboratory selected by the Contractor and detailed in the Contractor Submissions. Alternate laboratories utilized by the Contractor shall require approval by the ECC Trust and the United States Environmental Protection Agency/Indiana Department of Environmental Management (USEPA/IDEM) prior to performance of any analytical work.

Sand and Gravel Monitoring Wells

Subsurface water samples will be collected from sand and gravel monitoring wells S-1, S-4B, and S-5 in accordance with USEPA-approved low flow sampling procedures outlined in a November 10, 2000 letter, which is provided in Appendix C-3.

B. Surface Water Sampling Procedures

The surface water samples from Unnamed Ditch will be collected in accordance with the surface water sampling procedures outlined in the 1997 FSP and included as Appendix C-4.

C. Soil Vapor Sampling Procedures

Augmented SVE trench segment soil vapor samples for laboratory analysis will be collected from vapor sample ports at the SVE manifold in the Treatment Building into a laboratory-supplied Tedlar bag or SUMMA canister (all analytes except phenol) and using XAD sorbent tubes (for phenol) in general accordance with the procedures contained in Appendix C-5. When sampling into a Tedlar bag or the sorbent tubes, a vacuum sampling pump will be used to withdraw the sample from the vacuum system into the bag/tube.

Combined soil vapor samples from the untreated vapor stream will be taken from a sampling port after the SVE inlet pipe manifold in the Treatment Building. The procedures outlined in Appendix C-5 will be used for the combined sample collection also. The vapors from the augmented SVE trenches must be isolated from the vapors from the *ex situ* SVE soil treatment cell, if in use, prior to obtaining this combined sample. If SUMMA canisters are used, the vacuum in the canister is expected be sufficient to draw in the sample. A vacuum sampling pump will be used when Tedlar bags or sorbent tubes are used to collect the samples.

Confirmation soil vapor samples taken after the 21 day shutdown of the system will be collected as follows. The SVE system will be restarted for approximately 30 minutes to purge the SVE collection piping, then will be shut down again. At each SVE wellhead, the SVE inlet port will be connected to a vacuum sampling pump and the line will be purged for approximately 2 to 3 minutes. The tubing will then be attached to a 6-liter Summa canister. The regulator of the Summa canister will be laboratory-set for a 15 to 30 minutes sampling period. At the end of the sampling period (or when approximately 5 inches mercury vacuum remains in the canister), the sampling will be complete and the canister inlet will be closed. The sample canister will be handled as described in Appendix C-5. The Summa canister samples will be analyzed for the VOC and SVOC parameters listed in Table 2-2 of the Design Report. Upon completion of the Summa canister sampling, the sample for phenol analysis will be obtained from the same SVE wellhead port using the vacuum sampling pump and XAD sorbent tubes as described in Appendix C-5.

Any additional vapor samples collected from the *ex situ* SVE soil treatment cell will be collected using the same procedures as vapor samples collected during operation of the augmented

SVE system. The vapor from the *ex situ* SVE soil treatment cell must be isolated from the vapors from the augmented SVE trenches for this sampling.

D. Soil Waste Characterization Sampling Procedures

One soil waste characterization sample will be collected from each stockpile of excess soil excavated from the augmented SVE trenches in accordance with the procedures contained in Appendix C-6. The samples will be collected as grab samples from a depth of approximately one foot below the surface of the stockpile.

E. Trench Water Biopolymer Breakdown Sampling Procedures

Samples of trench water and containerized biopolymer slurry following the enzyme treatment will be collected as grab or composite samples from the trenches in accordance with procedures for water sampling using dedicated bailers and described in Appendix C-2.

F. PRGS Effluent Monitoring Procedures

Sampling of PRGS effluent will be conducted after the conclusion of the 2-years Phase II(a) Long-Term Monitoring period using a dedicated Teflon bailer as described for subsurface water sampling in Appendix C-2. Water will not be evacuated from the PRGS treatment vessel sample port prior to sample collection.

G. Wastewater Discharge Monitoring Procedures

The sample of the treated wastewater will be collected from Tank T-4 using a dedicated disposable bailer. Since Tank T-4 is not covered, the bailer may be lowered from the access stairway.

H. Water Level Measurements

Water level measurements will be collected from the TBCW piezometers and SVE trench dewatering wells according to the procedures contained in Appendix C-2.

I. Sample Designation

A sample numbering system has been developed for the Attachment Z-1 Remedy that will include the following sequential information:

- Name of Site – ECC;
- Sample Matrix – Soil Vapor (SV); Subsurface Water (SSW), Combined Subsurface Water (CSSW), Surface Water (SW), Soil (S); PRGS Effluent (EFF), and Augmented SVE System Wastewater (WW);
- Soil, soil vapor, trench segment, monitoring well, surface water location, trip blank number, or other appropriate location identifier;
- Sample date; and
- Quality Assurance/Quality Control (QA/QC) Modifiers – Field equipment blank (B), field duplicate (D), and matrix spike/matrix spike duplicate (M).

For example, a sample collected from Trench Segment 2 during a subsurface water sampling event on October 2, 2008 would be labeled ECCSSW-TS2-100208. All field samples will be identified with sample identification labels consisting of gummed paper labels that include the sample designation and the following additional information:

- Site name
- Project number
- Name of collector
- Affiliation of collector
- Date and time of collection
- Analyses requested

J. Decontamination Protocol for Sampling Equipment

The decontamination protocol for sampling equipment is provided in Appendix C-7.

K. Sample Handling and Analysis

Sample handling and analytical procedures are presented in Appendix C-8.

TABLES

TABLE C-1

Sampling Schedule and Analytical Parameters
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix Sampled	Sampling Area	Frequency	Field Parameters	Laboratory Analyses	Laboratory Methods	Duplicates	Trip Blanks	Field Equipment Blanks	MS/MSD
Soil Vapor- Individual Trench Segments	SVE System- Treatment Building at field piping connection to building piping	Daily- 1st week, weekly- next 4 weeks, biweekly thereafter during Active Phase	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor- All Trench Segments Combined	SVE System- Treatment Building at the in-line analyzer	Daily for first 5 weekdays, thereafter as required by Contractor for system optimization, if correlation established	Total organics using in-line Continuous Analyzer, Vapor flow rate	total organics	Total Organics- Field Series 8800 Continuous Analyzer	--	--	--	--
Soil Vapor- All Trench Segments Combined	SVE System- Treatment Building at manifold samling port	Daily for first 5 weekdays	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor - Individual trench segments	SVE well head at trench segment	Following restart for confirmaton of shut down criteria	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor- *Ex-situ* Treatment Cell, Combined	SVE System- Treatment Building	Minimum of one sample per month during operation of SVE for *ex-situ* treatment cell and following *restart spike tests to confirm* shutdown	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	
Excess Soil Excavated from Augmented SVE Trenches	Soil stockpiles from trench excavation	One soil sample per augmented SVE trench	PID	VOCs, SVOCs, Inorganics, and PCBs by SPLP and non-leachate analysis of VOCs and SVOCs[2]	SW-846 Methods 8260B, 8270C, 6010, and 8082	--	--	--	--
Water (biopolymer slurry after enzyme addition)	*Biopolymer Slurry from SVE* trench segments	One-time	Field tests as required to confirm biopolymer slurry breakdown	BOD, viscosity, and/or other parameters to confirm biopolymer slurry breakdown	SW-846 Method 405.1 for BOD; appropriate physical test methods for viscosity or other parameters to confirm biopolymer slurry breakdown	--	--	--	--
Subsurface Water- single combined water sample[3]	PRGS Pipe Collection Manhole	One sample following two successful Restart Spike Tests and one sample following each 90 days of additional SVE system operation	pH, specific conductance, temperature *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, 1LM04.1	1/10	1/ shipment	1/10	--

TABLE C-1

Sampling Schedule and Analytical Parameters
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix Sampled	Sampling Area	Frequency	Field Parameters	Laboratory Analyses	Laboratory Methods	Duplicates	Trip Blanks	Field Equipment Blanks	MS/MSD
Subsurface water within each trench segment	Augmented SVE Trench Dewatering Wells	Semi-annual sampling of each operating trench segment during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods[5]	pH, specific conductance, temperature, water level *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Treated Subsurface Water/Effluent	PRGS Treatment Vessel-Effluent Sampling Port	Annual sampling after completion of the 2-year of Phase II(a) monitoring	pH, specific conductance, temperature *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Subsurface Water	Sand and Gravel Monitoring Wells S-1, S-4B and S-5	Semi-annual sampling during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods	pH, specific conductance, temperature, water level *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Surface Water	Unnamed Ditch, at Locations SW-1, NSL-1 and SW-2	Semi-annual sampling during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods	Stream Observations	VOCs, SVOCs[6] some metals, cyanide	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	--	1/20
---	Thin Barrier Curtain Wall Piezometers	Quarterly during the 1-year Phase I and 2-year Phase II(a) monitoring periods	Water Level Measurements	--	--	--	--	--	--
Augmented SVE System	Wastewater Discharge Monitoring- Tank T-4 or discharge port	Prior to each batch discharge	--	VOCs with Approved Effluent Limits (see Attachment C-1 of FSP Addendum)	SW-846 Method 8260B	1/10	--	1/10	N/A

Key: VOCs= volatile organic compounds; SVOCs= semivolatile organic compounds; PCBs= polychlorinated biphenyls
SVE= soil vapor extraction
PID = Photoionization Detector
MS/MSD = Matrix Spike/Matrix Spike Duplicate
-- = None/Not Applicable
TBD = To be determined

[1] VOCs and SVOCs listed in Table 3-1 with Soil Vapor Standards. (Table 3-1 in the Design Report for the Attachment Z-1 Remedy)

[2] Analysis of VOCs and SVOCs with Site Specific Soil Exposure Concentrations in Table 2-2 of the Design Report for the Attachment Z-1 Remedy; additional analysis may be added if disposed offsite.

[3] If no water in PRGS system at end of Active Phase, sampling will be considered complete.

[4] VOCs and SVOCs listed in Table 2-1 with Acceptable Stream Concentrations.

[5] Additional samples may be collected during SVE system operation at the discretion of the ECC Site Trust.

[6] VOCs and SVOCs listed in Table 2-1 with Acceptable Surface Water Concentrations (Table 2-1 in the Design Report for the Attachment Z-1 Remedy)

TABLE C-2

Field Equipment
Enviro-Chem Superfund Site
Zionsville, Indiana

Activity	Field Equipment
Subsurface Water Monitoring- Augmented SVE Trenches	Teflon bailers/cord Water quality meter Water level indicator PID PPE
Subsurface Water Monitoring- Sand and Gravel Wells S-1, S-4B and S-5	Well Pumps and tubing Flow-through cell and water quality meter Water level indicator Drums for temporary containment of purge water PID PPE
Subsurface Water Monitoring- PRGS Collection Manhole	Laboratory Cleaned Sample Collection Bottle Teflon bailer/cord PID PPE
PRGS Effluent Monitoring	Teflon bailer/cord Water quality meter Water level indicator PID PPE
Surface Water Sampling	Laboratory Cleaned Sample Collection Bottle Marking stakes to mark sampling station PPE
Soil Vapor Sampling (for laboratory analysis)	Laboratory-prepared Summa cannisters with flow controllers or Tedlar bags, sampling pump, XAD sorbent tubes, ancillary equipment
Soil Waste Characterization Sampling	Marking flags/stakes Stainless steel sample spoons/scoops PID PPE
Trench Water Biopolymer Break Down Sampling	Bailers, pump or other sample collection tools Field analytical test kits, as necessary
Decontamination	Alconox™ Brushes Distilled Water Paper Towels Tap Water Water containers PPE Drums for temporary containment of decon water
Miscellaneous	Calibration equipment for PID meter *Field book* Sample coolers, chain-of-custody forms, sample containers, lables and custody seals Packing and shipping material Field forms and logs Permanent markers

Key:
PPE = Personnel protective Equipment
PID = Photoionization dector

TABLE C-3

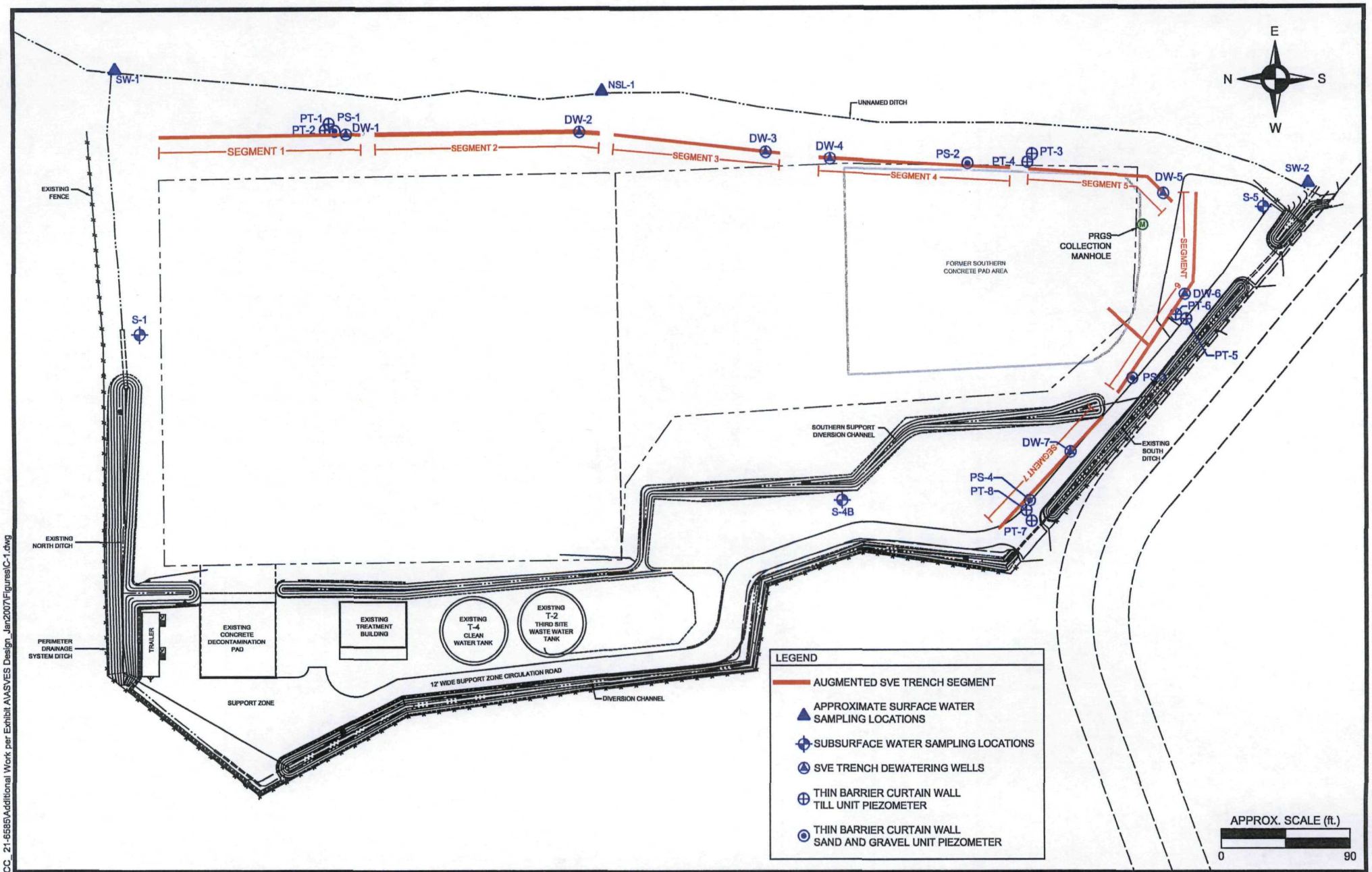
Sampling Containers, Preservation, and Holding Times
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix	Parameter	Container	Preservation	Hold Times	Sample Volume
Water	VOCs	Glass Vials	HCl to pH <2; Cool to 4 °C	14 days	3 x 40 mL
Water	SVOCs	Amber Glass	Cool to 4 °C	7 days to extractions; 40 days until analysis	2 X 1000 mL
Water	some metals and cyanide[1]	Plastic	NaOH	14 days	500 ml
Soil	VOCs (SPLP)	Glass	Cool to 4 °C	14 days to leach; 14 days from leach to analysis	4 oz.
Soil	VOCs	Glass Vials[2]	Cool to 4 oC 2 with 5 mL sodium bisulfate, 1 with 5 mL methanol	14 days	3 x 40 mL
Soil	SVOCs	Clear Glass	Cool to 4 °C	14 days to extractions 40 days until analysis	8 oz.
Soil	SVOCs, PCBs (SPLP)	Clear Glass	Cool to 4 °C	14 days to leach; 7 days from leach to analysis	8 oz.
Soil	Metals (not including mercury) (SPLP)	Clear Glass	Cool to 4 °C	180 days to leach; 180 days from leach to analysis	8 oz.
Soil Vapor	VOCs, 1,2-Dichlorobenzene	SUMMA Canister or Tedlar Bag	NA	7 days	1000 mL or 6000 mL
Soil Vapor	Phenol	XAD sorbent tubes	Cool to 4 °C	7 days	XAD sorbent tubes

Key:

VOCs = Volatile organic compounds
SVOCs = Semivolatile organic compounds
HCl = Hydrochloric acid
PCBs = Polychlorinated biphenyls
SPLP = Synthic Precipitation Leaching Procedure
Note 1: Analysis list from Table 2-1 of the Design Report for the Attachment Z-1 Remedy. Acceptable Stream Concentration list.
Note 2: Encore samples may be used instad of set of glas vilas if desired, however hold-time decreases.

FIGURE



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ENVIRON

SURFACE AND SUBSURFACE WATER SAMPLING LOCATIONS
ENVIRO-CHEM SUPERFUND SITE
985 S. US. HIGHWAY 421
ZIONSVILLE, INDIANA

Figure
C-1

Drafter:	APR	Date:	01/22/07	Contract Number:	21-6585K	Approved:	Revised:

APPENDIX C-1

Wastewater Effluent Limits
February 27, 1997

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
We make Indiana a cleaner, healthier place to live

Frank O'Bannon
Governor

Michael O'Connor
Commissioner

100 North Senate Avenue
P.O. Box 6015
Indianapolis, Indiana 46206-6015
Telephone: 317-232-8603
Environmental Helpline 1-800-451-6027

February 27, 1997

Mr. Norman Bernstein
N.W. Bernstein & Associates
2000 M Street N.W., Suite 745
Washington, D.C. 20036

Dear Mr. Bernstein:

Re: NPDES Effluent Limits for the
Enviro-Chem Superfund Site

The Indiana Department of Environmental Management, Office of Water Management (IDEM-OWM) has issued the final effluent limitations for the discharge of wastewater from the remedial action to be taken at the Enviro-Chem site. The final effluent limits and a briefing memo explaining the treatment system to be used on-site, the monitoring requirements to ensure compliance with the effluent limits, and the rationale used to determine the effluent limits are included with this letter.

If you have any questions about the enclosed information, please contact me at (317) 308-3120.

Sincerely,

Tony Likins

Tony Likins
Superfund Section

AWL/tl
Enclosure
cc: Mike McAteer, U.S. EPA Region 5
Catherine Gibbs, IDEM-OLC

cc: (w/o enclosure)
George Oliver, IDEM-OWM
Steve Roush, IDEM-OWM
Pat Carrasquero, IDEM-OER

MAR 3 1997

ENVIRO-CHEM SUPER FUND SITE			Indiana Department of Environmental Management				Pg 1 of 2
ZIONSVILLE, INDIANA, BOONE COUNTY			Office of Water				
Effluent Limits FINAL			Feb. 20, 1997				
Chemicals	PROPOSED TREATMENT	MCL's	NWQ	RREL BPJ/BAT	TECHNOLOGY TYPE	MOST STRINGENT	FINAL LIMITS
	ug/l	mg/l	mg/l	mg/l		mg/l	mg/l
Volitile Organics							
1,1-Dichloroethylene	1.85	0.007	0.033(DM)	0.001(98.6%)	AirS	0.002	0.002
1,2-Dichloroethene	1.85	NA	NA	NA		0.002	0.002
Ethylbenzene	3,200	0.7	1029(DM)	0.001(90%)	AirS	0.002	0.7
Dichloromethane(Methylene Cl2)	15.7	0.005	0.819(DM)	0.001(99.6%)	AirS/GAC	0.001	0.005
Tetrachloroethylene	8.85	0.005	0.145(DM)	0.001(99%)	AirS	0.001	0.005
Toluene	3400	1	0.476(DM)	0.001(97 & 99%)	AirS/GAC	0.001	0.48
1,1,1-Trichloroethane	202	NA	0.254(DM)	0.001(99%)	AirS/GAC	0.001	0.2
1,1,2-Trichloroethane	41.8	NA	0.662(DM)/0.006(HH)	NA	NA	0.042	0.042
Trichloroethylene	69.7	0.005	0.163(DM)	0.001(99.5%)	AirS	0.001	0.01
Vinyl Chloride	525	0.002	6817(DM)	0.001(99.8%)	AirS	0.001	0.01

ENVIRO-CHEM SUPER FUND SITE							
ZIONSVILLE, INDIAN, BOONE COUNTY			Feb. 20, 1987				
Effluent Limits FINAL							
Chemicals	PROPOSED TREATMENT	MCL's	WQ	RREL BPJ/BAT	TECHNOLOGY TYPE	MOST STRINGENT	FINAL LIMITS
	ug/l	mg/l	mg/l	mg/l		mg/l	mg/l
Semivolatile Organics							
bis(2-Ethylhexyl)phthalate	594	NA	0.591(DM)/0.360(AC)	NA	NA		0.59
di-n-Butylphthalate	3,447	NA	0.021(DM)/0.013(AC)	NA	NA	0.021	0.021
Diethylphthalate	7,076	NA	2957(DM)	NA	NA		7.
1,2-Dichlorobenzene	763	NA	4.27(DM)	0.020(80%)	AIS	0.02	0.76
Naphthalene	620	NA	0.069(DM)		0.069/40CFR	0.059	0.069
Phenol	570	NA	1.0(DM)/0.569(AC)	NA	NA		0.57

APPENDIX C-2

Subsurface Water Sampling Procedures

One duplicate sample will be collected per group of 10 or fewer soil samples. Drilling augers will be steam cleaned between each sampling location, and split-spoon samplers will be steam cleaned and rinsed with distilled water between the collection of each sample. Any other equipment that comes into contact with a sample will be decontaminated as described in Table 6-1.

6.2.2 Borrow Area Soils

The borrow area soils slated for use in the final cover and as backfill for the southern concrete pad excavation will be sampled using a test pit operation procedure where an excavator or backhoe will dig from surface to the intended vertical limit of useable soils. Sampling will include discrete sampling of the soils throughout the vertical profile of the borrow area.

The limits of the useable soils will be determined by the geotechnical soil analysis (e.g. gradation, Atterberg limits, etc.) as specified in the Technical Specifications and as directed by Appendix A of the Construction Quality Assurance Plan (CQAP). The contractor will be responsible for determining the required number of samples based on the number of borrow areas and useable soils configuration (horizontal/vertical) in each. The final number of samples and analyses of borrow soils will be approved by the Engineer prior to the Contractor performing the sampling.

6.3 Subsurface Water Sampling

Samples from the subsurface wells will be collected semiannually during the operation of the SVE system (Soils Cleanup Verification Phase) and analyzed as specified in Section 4.3. Compliance monitoring will be continued ~~on a semiannual basis for 7 years after Soil Cleanup Verification is accomplished~~, as specified in ~~Section 4.0 of Exhibit A to the Consent Decree~~ Attachment 2-1.

Table 6-1. Decontamination Protocol for Sampling Equipment

Step Number	Description
1	Scrub equipment thoroughly with soft-bristled brushed in a low-suds detergent solution.
2	Rinse equipment with tap water by submerging and/or spraying.
3	Rinse equipment with methanol by spraying until dripping: retain drippings.
4	Rinse equipment with distilled water by spraying until dripping; retail drippings.
5	Rinse equipment with distilled water a second time by spraying until dripping; retain drippings.
6	Place equipment on plastic or aluminum foil and allow to air dry for 5 to 10 minutes.
7	Wrap equipment in aluminum foil (shiny side out) for handling and/or storage until next use.

6.3.1 Water Level Measurement

Static water levels will be measured to the nearest 0.01 foot in each monitoring well and the piezometer at each sampling event and recorded in the field notebook. The water level surface will be measured prior to well purging and sampling by using an electric water level meter. Before lowering the probe in the well, the batteries will be checked by pressing the test button on the instrument. The probe will be slowly lowered into the well until contact with the water surface is indicated on the meter. The probe will be withdrawn just above the water surface, and a second reading will be taken prior to withdrawing the probe from the well. Both readings will be recorded in the field logbook. The probe will be decontaminated prior to inserting the instrument into a well by washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water.

Each well will have a reference point, indicated on the inner well casing, from which water level measurements will be taken. The reference point elevation on the well will be

established by a survey with respect to U.S. Datum mean sea level elevation to an accuracy of 0.01 feet for computation of the subsurface water elevation.

6.3.2 Well Depth Measurement

The total depth of the well will be measured and recorded prior to well purging and sampling. A weight tied to a length of cotton cord will be used to tag the bottom of the well, and the length of cord used will be measured to establish well depth. The weight will be rinsed with distilled water and the cotton cord will be replaced between measurements.

6.3.3 Well Evacuation

Standing water in the wells will be removed prior to sampling by purging until: (1) at least three well volumes have been removed; (2) the well yields low turbidity water; and (3) consistent values of temperature, pH, and specific conductance are achieved. If the well goes dry before three well volumes have been removed, samples will be taken as soon as the well recovers. The calculation of well volume will be as follows:

- The well casing inside diameter will be measured;
- The static water level below the measuring point will be determined;
- The total depth of the well will be identified from the measuring point;
- The number of linear feet of static water will be calculated as the total depth of the well minus the static water level; and
- The static volume (well volume) will be calculated in gallons as:

$$V = (\pi r^2)(h)(7.48)$$

Where:

V = well volume (gal)

$\pi = 3.14$

r = well radius (ft)

h = linear feet of static water (ft)

Dedicated Teflon or stainless steel bailers will be used for purging and sampling the wells. Purged water will be placed in containers for subsequent handling and disposal in accordance with Federal, state, and local regulations based upon the results of chemical analysis. Bailers, pumps, and all other equipment shall be decontaminated prior to insertion into the well. Decontamination will consist of steam cleaning or washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water. Bailer ropes and sampling gloves will be discarded after sampling each well.

6.3.4 Groundwater Sampling

During sampling, special care will be taken to avoid physically altering or chemically contaminating the sample volumes. Sampling of onsite till wells will not occur until the SVE system has been shut down, and till waters have been given sufficient time to stabilize as described in Section 6.3.3.

Sampling will be performed with bottom-filling Teflon or stainless steel bailers. Subsurface water pH, specific conductance, and temperature will be determined in the field on secured samples. Sample volumes will be collected in the following order:

- Volatile organics;
- Base neutral/acid extractable organics;
- Polychlorinated biphenyls (PCBs);
- Metals; and
- Cyanide.

Samples of subsurface water will be prepared, preserved, and stored as described in Section 7.0. All sampling equipment will be decontaminated between samples following the procedures in Table 6-1.

The objective of the subsurface water sampling for the metals and PCBs shown in ~~Table 4-3~~ Table 2-1 of the Design Report for the Attachment 2-1 Remedy is to determine the concentration of dissolved constituents. Therefore, subsurface water

samples for metals and PCB analyses will be filtered through a nonmetallic 0.45-micron pore size membrane immediately after collection. One of the following apparatus will be used for field filtration: (1) a Sartorius filtration apparatus or (2) a Nalgene filtration apparatus. If necessary, the sample may be pumped through the filter using a Nalgene hand vacuum pump. The first 150 to 200 ml of filtrate will be used to rinse the filtration apparatus of any contaminants. This technique minimizes the risk of altering the composition of the samples by the filtering operation. The filtrate for metals analysis will be collected in a polyethylene bottle and immediately acidified to a pH <2 using nitric acid. The filtrate for chromium VI analysis will not be acidified. The filtrate for PCB analysis will be collected in amber glass bottles.

One field blank sample will be collected for each group of 10 or fewer samples. Equipment in safe blank samples will be prepared immediately after collection of a field sample by pouring distilled water through a decontaminated bailer into the appropriate sample container. Preparation of the field blank will occur onsite.

One field duplicate sample will be obtained for each group of 10 or fewer compliance samples.

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of one per group of 20 or fewer compliance samples designated for organics analysis.

Trip blank samples will be provided by the laboratory selected to perform volatile organic analysis at a frequency of one per shipping container of samples.

6.4 Surface Water

The surface water will be monitored by sampling the unnamed ditch just upstream and just downstream of the ECC Site (Figure 4-1). To collect a surface water sample, the sample container will be submerged in the water, removed, and immediately capped. The container mouth will be positioned so that it faces upstream, while the sampling personnel are standing downstream to prevent the stirring up of any sediments that would contaminate the sample. Downstream samples will be collected first moving upstream. Quality control samples (field blanks, field duplicates, and MS/MSD samples) will be collected at the same frequency as specified for subsurface water samples. Decontamination of sampling equipment will consist of

APPENDIX C-2 Addendum

Field Measurement of Ground Water Quality Parameters

A. Introduction

In accordance with the ground water sampling procedures in Appendix C-2 and Appendix C-3 of this FSP Addendum, measurements of ground water parameters will be performed during the well purge prior to sampling. A Horiba U-22 water quality meter, or equivalent, will be used to obtain pH, specific conductivity and temperature measurements periodically. The procedures for the U-22 equipment are outlined below. The specific meter manual should be consulted for troubleshooting, data storage and additional calibration methods.

B. Auto Calibration Procedures

The sensors should be calibrated before performing measurements. In the Auto-calibration mode, the pH and conductivity sensors are calibrated simultaneously.

- Wash the sensors in distilled water
- Immerse sensor into a beaker of pH4 standard solution (supplied with rental equipment).
- Turn the meter power on and press the MEAS button until pH is lit.
- Press the CAL key
- Press the ENT key to start Auto-calibration.
- Wait for END to be displayed, indicating the end of calibration.
- Press the MEAS key to return to measurement mode.
- If one of the parameter names continues to blink, an error has occurred. Consult the meter manual.
- Neutralize any basic pH4 fluids before disposal.

2. Measurement

Measurement may be performed with the sensors in a flow-through cell or another appropriate container. The sensors must be fully immersed for proper readings.

- Remove the protective sleeve and any other packaging and wash the probes in distilled water prior to use.
- Immerse the sensor in the sample.
- Use the MEAS key to switch measurements to the desired parameters.
- Record the reading and the units. Note that units might change from reading to the next.

3. Maintenance/Storage

After use, clean the sensor probe in tap water and wipe of contamination. Next, put distilled water in the calibration beaker to the marked line and attach to the sensor probe.

If contamination is not removed by the tap water rinse, see the equipment manual for cleaning the individual probe units.

APPENDIX C-3

Low-Flow Sampling Proposal
Dated November 10, 2000

ENVIRON

November 10, 2000

Via Facsimile and Federal Express

Mr. Michael McAteer
U.S.EPA, HSRW-6J
77 West Jackson Blvd.
Chicago, IL 60604-3590

Re: Low Flow Ground Water Sampling
ECC Superfund Site
Zionsville, Indiana

Dear Mr. McAteer:

The purpose of this letter is to propose changes in the methodology for the purging and sampling of the till wells at the ECC Superfund Site in Zionsville, Indiana. For the past six sampling events, the ground water samples from the till wells were collected as described in Section 6.3 of the Radian Revised Remedial Action Field Sampling Plan, Revision 4, dated April 28, 1998 (FSP). In accordance with the FSP, the till wells were purged of a minimum of three well volumes or until the wells went dry, prior to sampling. The water in the till monitoring wells was evacuated using dedicated polyethylene disposable bailers and sampled using dedicated Teflon disposable bailers.

As stated within each of the quarterly and semi-annually sampling reports, most of the till wells were purged (bailed) dry before the three well volumes could be removed. In addition, the purging and sampling with a bailer increased the turbidity of the purge and sample water. ENVIRON believes that sampling procedures that cause less disturbance, and therefore less turbidity, produce the most reproducible and representative samples.

In an effort to decrease the turbidity of the purge and sample water and to limit the number of wells that are being purged dry, ENVIRON is proposing low flow purge and sampling methods for all ten ECC till wells. The six off-site till wells would be purged and sampled using the same methodology as is used to purge and sample the sand and gravel wells. This method involves the use of a peristaltic pump and dedicated Teflon tubing to sample the wells after three well volumes had been purged with a peristaltic pump. The intake for the Teflon tubing would be placed at the bottom of the screened interval and the pump would be set to its lowest flow rate.

The four on-site till wells will be purged and sampled using dedicated PVC bladder pumps and dedicated Teflon tubing. Because the on-site till wells are screened at a greater depth below the present ground surface than the off-site till wells (due to the

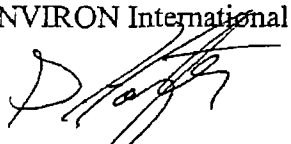
placement of the contaminated fill from the Southern Pad area as well as the placement of the RCRA cover in this area) the depth to the bottom of the screened interval is to great for the use of a peristaltic pump. In addition, the bladder pumps will provide a pump rate even lower than the peristaltic pumps, thus further decreasing the turbidity of the sample.

With your approval, ENVIRON expects to conduct the Fourth Quarter 2000 ground water sampling, using the above mentioned low flow sampling methods, during the week of November 27, 2000.

If you have any questions, please do not hesitate to contact us.

Sincerely,

ENVIRON International Corporation



Scott Hayter, P.G.
Senior Associate

SH

cc: Mr. Myron Waters – IDEM
Mr. Tim Harrison – CH2M Hill
Dr. Roy Ball – ENVIRON International Corporation
Mr. Norman Bernstein – N. W. Bernstein & Associates, L.L.C.

A P P E N D I X C-4

Surface Water Sampling Procedures

samples for metals and PCB analyses will be filtered through a nonmetallic 0.45-micron pore size membrane immediately after collection. One of the following apparatus will be used for field filtration: (1) a Sartorius filtration apparatus or (2) a Nalgene filtration apparatus. If necessary, the sample may be pumped through the filter using a Nalgene hand vacuum pump. The first 150 to 200 ml of filtrate will be used to rinse the filtration apparatus of any contaminants. This technique minimizes the risk of altering the composition of the samples by the filtering operation. The filtrate for metals analysis will be collected in a polyethylene bottle and immediately acidified to a pH <2 using nitric acid. The filtrate for chromium VI analysis will not be acidified. The filtrate for PCB analysis will be collected in amber glass bottles.

One field blank sample will be collected for each group of 10 or fewer samples. Equipment in safe blank samples will be prepared immediately after collection of a field sample by pouring distilled water through a decontaminated bailer into the appropriate sample container. Preparation of the field blank will occur onsite.

One field duplicate sample will be obtained for each group of 10 or fewer compliance samples.

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of one per group of 20 or fewer compliance samples designated for organics analysis.

Trip blank samples will be provided by the laboratory selected to perform volatile organic analysis at a frequency of one per shipping container of samples.

6.4 Surface Water

The surface water will be monitored by sampling the unnamed ditch just upstream and just downstream of the ECC Site (Figure C-1). To collect a surface water sample, the sample container will be submerged in the water, removed, and immediately capped. The container mouth will be positioned so that it faces upstream, while the sampling personnel are standing downstream to prevent the stirring up of any sediments that would contaminate the sample. Downstream samples will be collected first moving upstream. Quality control samples (field blanks, field duplicates, and MS/MSD samples) will be collected at the same frequency as specified for subsurface water samples. Decontamination of sampling equipment will consist of

washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water.

APPENDIX C-5

Soil Vapor Sample Collection Procedures

VERSAR INC.

Transmitted Via Facsimile

31 October 2000

Mr. Michael McAteer
USEPA
HSEW-6J
77 West Jackson Blvd.
Chicago, Illinois 60604-3590

Mr. Myron Waters
IDEM
100 North Senate Ave.
P.O. Box 6015
Indianapolis, IN 46206-6015

Re: Revised Remedial Action (RRA) at the Enviro-Chem Site, Zionsville, Indiana Soil Vapor Extraction System Sampling Protocol (SVESSP)

Dear Mr. McAteer:

On September 20, 2000, Versar, Inc. (Versar) participated in a conference call with the U.S. Environmental Protection Agency (USEPA) and CH2M Hill, Inc. (CH2M Hill) to discuss the Soil Vapor Extraction System Sampling Procedures (SVESSP) and requirements for the Enviro-Chem site in Zionsville, Indiana. During the call the following items were discussed:

- The SVESSP modified by Versar from the protocol established in the 100% RRA Final Design Report
- A second round of Soil Vapor Extraction (SVE) sampling data required to confirm that individual trench sampling does not exceed the criteria identified in Table 4.1, Revised Exhibit A
- Restart spike sampling
- Soil sampling after the restart spike sampling is completed

Modified SVESSP

The vapor sampling procedure identified in the 100% RRA Final Design Report (100% RRAFDR) Volume 2, QAPP/Field Sampling Plan, Section 6.1, *Extracted Soil Vapor* (applicable pages attached) for operational control of the SVE system was not implementable due to operational constraints. The operational constraints resulted from the high vacuum pressure (i.e., 10 inches of Hg) that exists in the SVE piping manifolds. The high vacuum pressure cannot be overcome with the specified personal sampling pump (i.e., maximum vacuum pressure of 2.2 inches of Hg).

After discussion with USEPA, IDEM, and CH2M Hill, Versar implemented a modified procedure for the routine collection of vapor samples for the purposes of operational monitoring. This procedure has been used for more than 20 months. An overview of the procedure follows:

- Pump-out any standing water in the manifold system and continuously operate the system for at least 48 hours prior to any sampling
- Determine the air flow rate at the manifold sampling port utilizing a Dwyer thermal anemometer
- Collect a vapor sample in a 12-liter Tedlar bag utilizing a high vacuum pump
- Attach a personal sampling pump to the Tedlar bag and follow the procedure identified in the 100% RRAFDR, Volume 2, QAPP/Field Sampling Plan, Section 6.1, *Extracted Soil Vapor*

The use of the high vacuum pump to extract the vapor sample overcomes the operational constraints while utilizing the remainder of the procedure as specified in the 100% RRAFDR Volume 2, QAPP/Field Sampling Plan, Section 6.1, *Extracted Soil Vapor.* Attached is a Proposed Revision to Section 6.1 that details the procedure.

Second Round of SVE Sampling

Revised Exhibit A requires a second confirmatory round of sampling from all trenches. This sampling event was performed during the week of 2 October 2000. The results of this second round of sampling are being transmitted under separate cover. The results confirm the data obtained for the first round of sampling, i.e., that all contaminant concentrations are below those specified in the Soil Vapor Concentrations In Equilibrium With Acceptable Soil Concentrations, Table 4-1, Revised Exhibit A. Therefore, the system is now ready for the restart spike testing program.

Modification to Restart Spike Sampling

The discussion focused on the specific location in the SVE system to use for collecting vapor samples and the techniques that will be used for sampling. Locations on both the inlet and discharge sides of the vacuum pump were considered. Subsequently, it was determined that the ambient air bleed at the inlet of the pump cannot be closed without causing the pump to overheating during the five-hour sampling period. If the bleed air valve remains open, the discharge vapor stream will be diluted, yielding erroneous data. Therefore, Versar recommends that the restart spike sampling be performed as follows:

- Locate the sampling point on the inlet side of the vacuum pump before the bleed air is admitted into the system
- Collect vapors directly onto the sorbent by using a high vacuum sampling pump
- Measure the flow rate using a mass flow meter that measures air flow independent of pressure and temperature

~~The attached Proposed Revision to Section 6.1 details this procedure~~.

Soil Sampling after the Restart Spike Sampling

USEPA and CH2M Hill agree that the vapor sampling of the trenches and the restart spikes themselves are solely indicators of the equilibrium that exists between the vapors and the soil. The required soil sampling after the restart spike sampling will ultimately determine whether the soil in SVE Treatment area has been remediated. USEPA/CH2M Hill will be selecting the location of soil samples to satisfy two objectives: (1) sampling of the entire SVE treatment area; and (2) hot spot evaluation based upon the operational vapor analysis performed earlier by Versar.

Versar should have the second round of SVE vapor sampling data available during the week of 30 October 2000 and will formally request that the restart spike sampling be scheduled with distribution of this data.

Please contact me at (215) 788-7844, extension 222 should you have any comments or require further clarification of the restart spike sampling procedures.

Very truly yours,

Charles J Gaffney
for

G. J. Anastos, Ph.D., PE
Project Manager

Attachments

cc: D. Ashline
R.O. Ball
N.W. Bernstein
C. Gaffney
T. Harrison

ENVIRO-CHEM
SVE Vapor Sampling Procedures and Equipment

Reference Document:

The USEPA approved 100% RRA Final Design Report, Volume 2, QAPP/Field Sampling Plan, Revision 4, Section 6.0 Sampling Procedures and Equipment

PROPOSED REVISION

6.1 Extracted Soil Vapor

Soil vapor samples will be collected from the combined vapor flow prior to entering the activated carbon system and from individual manifolds or extraction well laterals for volatile organic compound (VOC) analysis as follows:

- A high vacuum sampling pump will be attached to the sampling tap installed in the SVE system. A ~~12-liter~~ Tedlar bag will be connected to the discharge side of the sampling pump. Somma canister
- The sample tap valve will be opened until the Tedlar bag is filled with vapor. (Somma canister using procedures guidance provided by the laboratory (included in Appendix C-5).
- The sampling tap valve will be closed at the end of the sampling interval.
- ~~A calibrated personal sampling pump will be used to transfer vapor from the Tedlar bag through the carbon sorbent NIOSH tubes.~~
- The volume of vapor required ~~(10 liters)~~ to achieve the method detection limit will be pumped at a flow rate of 0.2 liters per minute ~~for a total of 50 minutes~~.
- ~~The activated charcoal tubes will be removed, capped, placed in sealable plastic "whirl pak" bags (as supplied by the selected analytical laboratory), and labeled.~~ Tedlar bag/summa canister
- The ~~tubes~~ will be carefully packed into new, clean ~~paint cans with loose charcoal in the bottom~~, which will then be stored in a cooled container (container), separate from other types of environmental samples.

The phenol vapor samples will be collected as follows:

- A high vacuum sampling pump will be attached to the sampling tap installed in the SVE system. A ~~12-liter~~ Tedlar bag will be connected to the discharge side of the sampling pump.
- The sample tap valve will be opened until the Tedlar bag is filled with vapor.
- The sampling tap valve will be closed at the end of the sampling interval.
- A calibrated personal sampling pump will be used to transfer vapor from the Tedlar bag through the XAD-~~4~~ sorbent ~~NIOSH~~ tube.
- The volume of vapor required ~~(10 liters)~~ to achieve the method detection limit will be pumped at a flow rate of 0.1 liters per minute ~~for a total of 100 minutes~~.
- The activated XAD-~~4~~ tube will be removed, capped, placed in sealable plastic "whirl pak" bag (as supplied by the selected analytical laboratory), and labeled.
- The tubes will be carefully packed ~~into new, clean paint cans with loose charcoal in the bottom, which~~ will then be stored in a cooled container, separate from other types of environmental samples.

~~The soil vapor sampling procedures for VOCs and phenol analyses will be modified during the restart spike events by using a flow rate of 0.04 liters per minute for a total of 5 hours, starting 30 minutes after restarting the SVE system, as specified in Section 4.2.1 of Exhibit A. The procedures for collecting samples will be as follows (see attached Figure)~~:

- The sampling port for collecting SVE vapors will be located in the vacuum piping downstream of the air/water separator, upstream of the vacuum pump, prior to the bleed air inlet point, and sufficiently distant from piping elbows.
- The sampling system will be connected to an isolation valve at the sampling port and will split the flow into two trains, one for the carbon sorbent (for VOCs) and one for the XAD-~~4~~ sorbent (for phenols). Each train will contain, in series, an isolation valve, sorbent tube(s), electronic mass flow meter, and a precision needle valve for calibrating flow.
- The two sampling trains will be connected to a pulsation-dampening chamber that will be evacuated by a diaphragm-type high vacuum sampling pump capable of producing vacuum pressure below 10 inches Hg vacuum.

- Prior to and again after each sampling event, the flow meter will be calibrated using a "Gilibrator" primary flow calibration device. The flow meter in each train will be calibrated at 0.04 liters per minute with a duplicate sorbent tube located between the calibrator and the flow meter. The flow rate will be set by adjusting the needle valve located between the flow meter and the vacuum source.

- The restart spike method requires that combined vapor samples be collected after each of four consecutive restart spikes, once every two weeks. Each restart spike requires shutting down the SVE system for three days. Then, beginning 30-minutes after restarting the SVE system, samples are collected using a flow rate of 0.04 liters per minute for five hours (total of 12 liters).

- To initiate a five hour sampling event, the sampling pump is started with the isolation valve closed and the pressure allowed to stabilize at a level that is lower than the SVE manifold pressure. The timing begins when the isolation valve is opened, drawing vapors through the sorbent tubes at the set flow rate.

- At five-minute intervals, the flow rate, sampling pump pressure, and SVE manifold pressure will be recorded. Adjustments to the needle valve will be made, if needed, to maintain the correct flow rate.

- The activated sorbent tubes will be removed after five hours, capped, placed in sealable plastic "whirl pak" bag (as supplied by the selected analytical laboratory), and labeled.

- The tubes will be carefully packed into new, clean paint cans with loose charcoal in the bottom, which will then be stored in a cooled container, separate from other types of environmental samples.

Decontamination of the vapor sampling equipment will be conducted prior to any sampling and between sampling events by purging the sampling train (except sorbent tubes) ~~with nitrogen~~ to remove any residual extracted soil vapor. Interconnecting tubing will be disposed of after each sampling event.

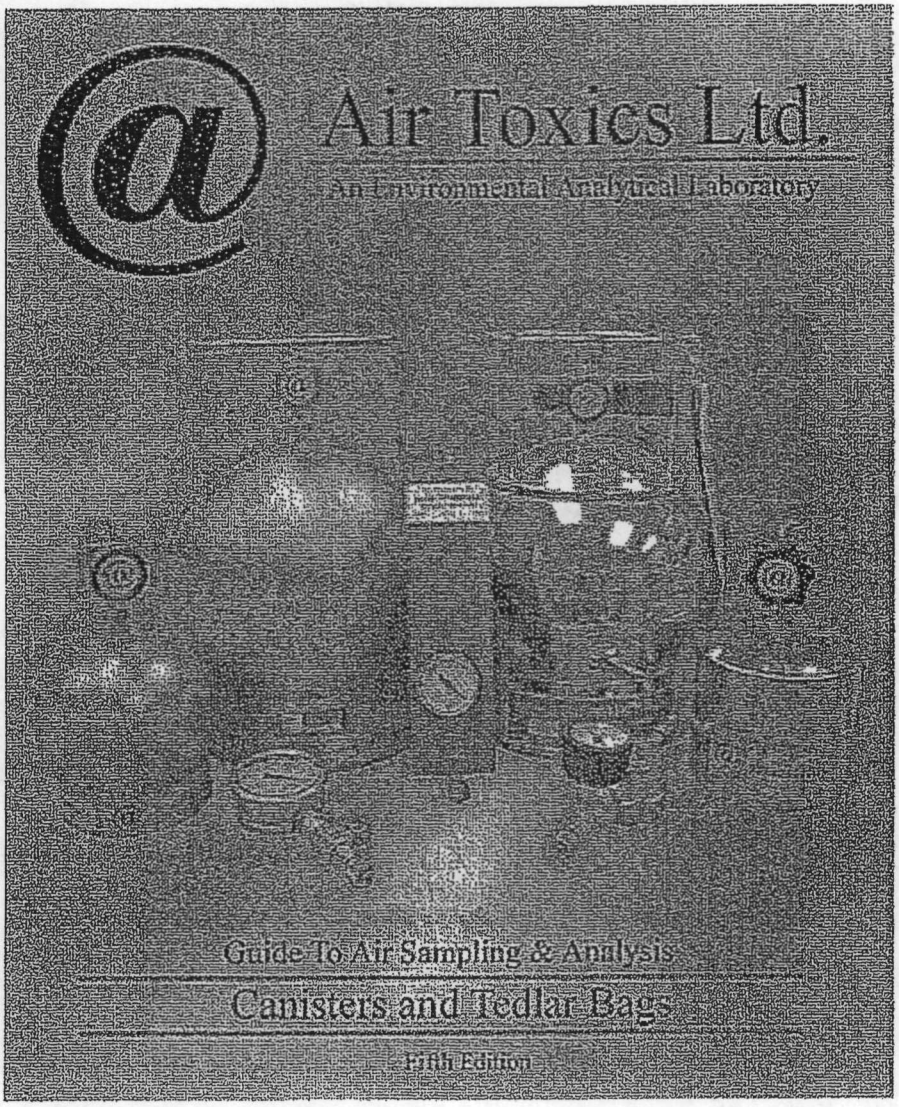

Air Toxics Ltd.
An Environmental Analytical Laboratory
Guide To Air Sampling & Analysis
Canisters and Tedlar Bags
Fifth Edition

Table of Contents

Tables

Section 1.0 Introduction

Air Toxics Ltd. presents this guide as a resource for individuals engaged in air sampling. Air sampling can be more involved than water or soil sampling due to the reactivity of chemical compounds in the gas matrix and sample interaction with the sampling equipment and media. Ensuring that air samples are collected properly is an important step in acquiring meaningful analytical results. This guide is not a substitute for experience and cannot sufficiently address the multitude of field conditions. Note that this guide is intended for projects involving whole air sampling of volatile organic compounds (VOCs) in canisters and Tedlar bags. Air Toxics Ltd. provides the "Guide to Air Sampling and Analysis - Sorbents, Solutions, and Filters" for other types of sampling.

1.1 Whole Air Sampling of VOCs

There are four general ways to collect compounds in a gas phase sample. A sampler can collect the gas in a container or draw the gas through a sorbent, solution, or filter. This guide focuses on collecting a sample in the most common air sampling containers, Summa canisters and Tedlar bags. The sample can be collected in the container either passively (i.e., by evacuating the canister prior to sampling) or actively (i.e., using a pump). The container is subsequently sealed and transported to the laboratory for analysis. The sample is referred to as a "whole air sample" and the compounds remain in the gas matrix (e.g., ambient air) inside the container.

As a general rule, whole air sampling is best when target compounds are chemically stable and have vapor pressures greater than 0.1 torr at 25deg and 760mm Hg, although exceptions to this rule can be found. Recovery of any given compound in a whole air sample is very much dependent upon the humidity of the sample, the chemical activity of the sample matrix, and the degree of inertness of the container.

1.2 Choosing Between Canisters and Tedlar Bags

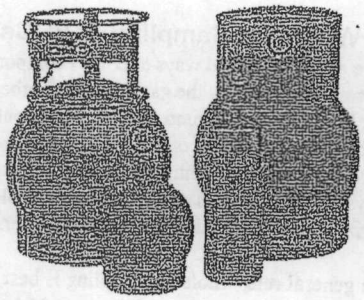
Table 1.2 compares the features of canisters and Tedlar bags. Canisters have superior inertness, hold time to analysis and ruggedness. They also do not require a sampling pump. Tedlar bags can be purchased inexpensively in bulk, carried to a sampling site in a briefcase, filled in seconds, and shipped easily to the laboratory for analysis. Call Client Services at 800-985-5955 if you have questions regarding sampling media.

Section 2. Canisters and Associated Media

This section provides a description of air sampling canisters, practical considerations for sampling, and step-by-step instructions for collecting a grab and integrated sample. Photographs illustrate the correct way to assemble the various sampling components. Tables provide detailed information on many operational factors that ultimately influence the quality of the data obtained from a canister sample.

2.1 Introduction to Canisters

An air sampling canister is a container for collecting a whole air sample for ambient and indoor air applications. A canister can be spherical or cylindrical and is constructed of stainless steel. The canister is prepared for sampling by evacuating the contents to a vacuum of approximately 29.9 inches of Mercury (in. Hg). Opening the stainless steel bellows valve allows the air sample to enter the canister. When the target volume of sample is collected, the valve is closed and the canister is returned to the laboratory.



Canisters can range in volume from less than 1 liter (L) to greater than 6 L. At Air Toxics Ltd., 6 L canisters are used for ambient air samples and for taking integrated samples. 1 L canisters are normally used for taking high concentration (i.e., greater than 5 ppbv) grab samples, although exceptions to these guidelines are common.

2.1.1 Summa Canister

A Summa canister is a stainless steel container that has had the internal surfaces specially passivated using a "Summa" process. This process combines an electropolishing step with a chemical deactivation step to produce a surface that is nearly chemically inert. A Summa surface has the appearance of a mirror: bright, shiny, and smooth. The degree of chemical inertness of a whole air sample container is crucial to minimizing reactions with the sample and maximizing recovery of target compounds from the container. Air Toxics Ltd. maintains a large inventory of Summa canisters in 6 and 1 L volumes.

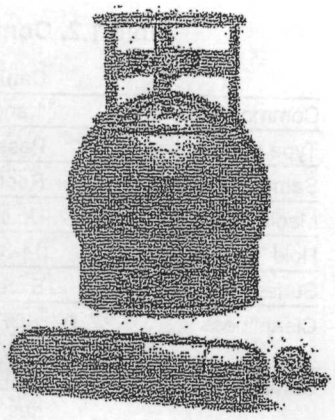
2.1.2 Canister Cleaning

Air Toxics Ltd. provides two types of canister cleaning certification, 10% and 100%, depending upon the requirements of the project. The 10% certification process is appropriate for routine ambient air applications and high concentration applications such as soil vapor and landfill gas monitoring. The 10% certification process begins by cleaning canisters using a

Table 1.2. Comparison of Canisters to Tedlar Bags

	Canisters	Tedlar Bags
Common Volumes	1 and 6 L	1, 3, and 5 L
Type of Sampling	Passive (vacuum)	Active (pump required)
Sample Handling	Room temperature	Room temperature
Media Hold Time	Up to 30 days recommended	Indefinite
Hold Time to Analysis	14-30 days	3 days
Surface Inertness	Excellent	Fair
Cleanliness	10% or 100% certified to ppbv/pptv levels	Some VOCs present at 0.5 to 45 ppbv
Sampling Application	Ambient/indoor air, soil/landfill gas, stationary source	Ambient air (fixed gases only), soil/landfill gas, stationary source
Rule of Thumb	"ppbv device"	"ppmv device"
Advantages	Inertness, hold time, ruggedness, no pump	Purchase/shipping cost, availability, convenience

combination of dilution, heat, and high vacuum. After completing the cleaning steps, 10% of the canisters are certified each day. Canisters are certified for approximately 60 VOCs using GC/MS. The 10% certification process requires that target compound concentrations be below 0.2 ppbv using GC/MS analysis. Alternatively, the 100% certification (i.e., individual certification) process is appropriate for ambient and indoor air applications driven by risk assessment or litigation that require pptv (parts per trillion by volume) sensitivity. Similar to the 10% certification, the 100% certification also begins with the canister cleaning process. The difference with the 100% certification is that canisters are individually certified for a client-specific list of target compounds using GC/MS. The 100% certified canisters are shipped with analytical documentation demonstrating that they are free of the target compounds down to the project reporting limits. When sampling with certified media it is important to note that all media is certified as a train and must be sampled as such (ie. a particular flow controller goes with a particular canister).



⇨ ***Specify whether your project requires***
10% or 100% canister cleaning certification.

2.1.3 Canister Hold Time

Media Hold Time : Canister sampling differs considerably from collecting a water sample in a VOA vial or a soil sample in an amber jar in that the container (valued at over $450) is cleaned and reused. Air Toxics Ltd. requires that our canisters be returned within 14 days of receipt to effectively manage our inventory. Once a canister is cleaned, certified, and evacuated we recommend the canister be used for sample collection within 30 days. Over time, low-level (pptv) concentrations of typical VOCs may off-gas from the canister surface resulting in potential artifacts in the sample results.

Sample Hold Time : Although 30 days is the most commonly cited hold time for a canister sample, the hold time is compound specific. For example, compounds such as chloroform, benzene, and vinyl chloride are stable in a canister for at least 30 days. In fact, EPA Method TO-15 states: "Fortunately, under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations for after storage times of up to thirty days". However, some VOCs such as bis(Chloromethyl)ether degrade quickly and demonstrate low recovery even after 7 days. The standard VOC list reported by Air Toxics is stable up to 30 days after sample collection. Some projects require a more rigorous 14 day hold time.

2.2 Associated Canister Hardware

Associated hardware used with the canister includes the valve, brass cap, particulate filter, and vacuum gauge.

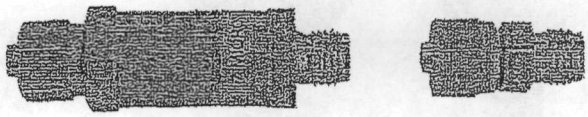
2.2.1 Valve

An industry standard, 1/4 in. stainless steel bellows valve (manufactured by Swagelok or Parker Instruments) is mounted at the top of the canister. The valve allows vacuum to be maintained in the canister prior to sampling and seals off the canister once the sample has been collected. No more than a half turn by hand is required to open the valve. Do not over-tighten the valve after sampling or it may become damaged. A damaged valve can leak and possibly compromise the sample. Some canisters have a metal cage near the top to protect the valve.

2.2.2 Brass Cap

Each canister comes with a brass cap (i.e., Swagelok 1/4 in. plug) secured to the inlet of the valve assembly. The cap serves two purposes. First, it ensures that there is no loss of vacuum due to a leaky valve or valve that is accidentally opened during handling. Second, it prevents dust and other particulate matter from fouling the valve. The cap is removed prior to sampling and replaced following sample collection.

⇨ **Always replace the brass cap following canister sampling.**



7 Micron 2 Micron

2.2.3 Particulate Filter

Particulate filters should always be used when sampling with a canister. Separate filters are provided to clients taking a grab sample. Filters are included in the flow controllers for clients taking integrated samples. Air Toxics Ltd. provides either a 2 micron filter or a 7 micron filter. These devices filter particulate matter greater than 2 and 7 micron in diameter respectively. The shorter 2 micron filter is a fritted stainless steel disk that has been pressed into a conventional Swagelok adapter and is disposed of after each single use. This device has a relatively high pressure drop across the fritted disk and restricts the flow into the canister. The 2 micron filter is standard for clients taking integrated samples. The longer 7 micron filter is cleaned in a similar manner as the stainless steel canisters after each single use and does not significantly restrict the flow rate into the canister. The 7 micron filter is primarily used with grab samples. Both the 2 and 7 micron filters are not calibrated devices and therefore the flow rates can and do vary for each filter.

⇨ **Always use the particulate filter for canister sampling.**

2.2.4 Vacuum Gauge

A vacuum gauge is used to measure the initial vacuum of the canister before sampling and the final vacuum upon completion. A gauge can also be used to monitor the fill rate of the canister when collecting an integrated sample. Air Toxics Ltd. provides 2 types of gauges. For grab sampling, a glycerine gauge is provided for checking initial and final vacuums only and is not to be sampled through. For integrated sampling a gauge is built into the flow controller and can be used for monitoring initial and final vacuums, as well as monitoring the fill rate of the canister. In special cases a pressure/vacuum gauge can be provided upon request. **Air Toxic Ltd's gauges are provided only to obtain a relative measure of "change." Individuals with work plans that outline specific gauge reading requirements are strongly encouraged to purchase and maintain their own gauges.**

⇨ **The gauges that Air Toxics Ltd. provides are for rough estimates only.**

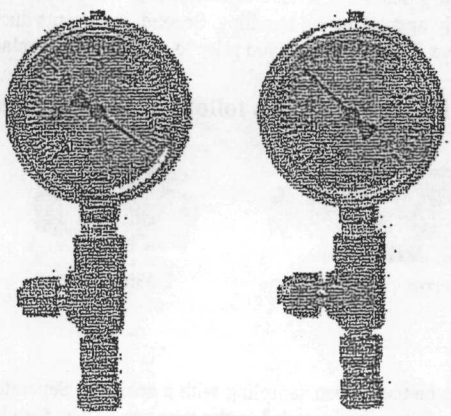


Table 2.2.3 Fill Times for Canisters

CANISTER VOLUME	7 micron filter	2 micron filter
6 L	16 sec	3 min
1 L	3 sec	30 sec

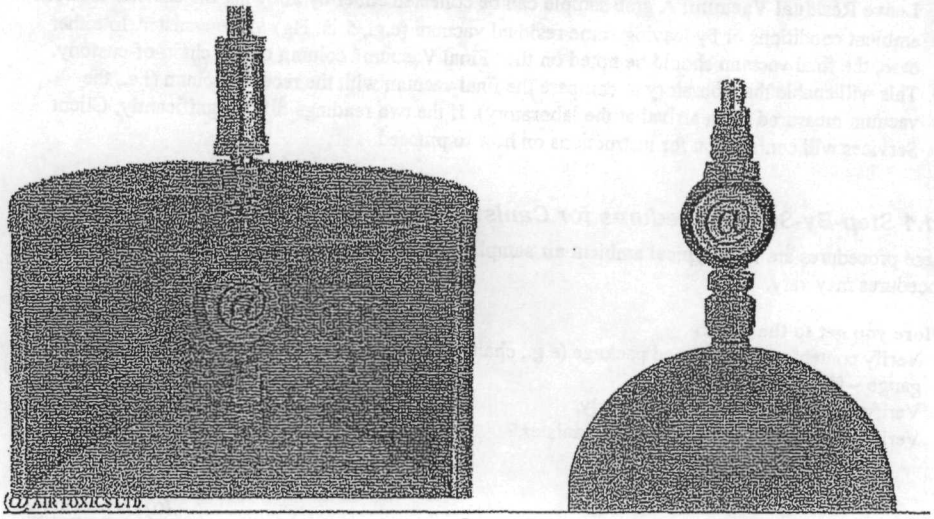
Section 3.0 Sampling with Canisters

There are two basic modes of canister sampling: grab and integrated. A grab sample is taken over a short interval (i.e., 1-5 minutes) while an integrated sample is taken over an extended period (e.g., 0.5-2 hours for a 1 L canister and 0.5-24 hours for a 6 L canister). In both modes the canister vacuum is used to draw sample into the canister. This is commonly referred to as passive sampling. Active sampling utilizes a pump to fill the canister. The most common hardware configuration used to take a grab sample are illustrated in the following figure. A particulate filter is used to prevent particulate matter from fouling the valve and entering the canister.

3.1 Considerations for Grab Sampling With Canisters

The following are some considerations for collecting a grab sample in a canister.

- **Avoid Leaks in Sampling Train:** All fittings on the sampling hardware are 1/4 in. Swagelok. A 9/16 in. wrench is used to assemble the hardware. It is not necessary to over tighten the fittings; finger tight plus 1/4 turn with the wrench is adequate. In practice this should be tight enough so



that the various pieces of equipment, when assembled, cannot be rotated by hand.

- **Verify Gauge Operation:** If the indicator does not read "zero" upon arrival, the gauge either needs to equilibrated or the gauge may be damaged and unusable. Equilibrate the gauge by "cracking" the rubber plug on top of the gauge. For more details on the equilibration procedure, see instructions included with the gauge or call Client Services at 800-985-5955.

- **Verify Initial Vacuum of Canister:** Prior to shipment, each canister is checked for mechanical integrity. However, it is still important to check the vacuum of the canister prior to use and record the initial vacuum on the chain-of-custody. The initial vacuum of the canister should be greater than 25 in. Hg. If the canister vacuum is less than 25 in. Hg, do not use it. Call Client Services at 800-985-5955 and arrange for a replacement canister. If sampling at altitude, there are special considerations for gauge readings and sampling (see Section 5.2). The procedure to verify the initial vacuum of a canister is simple, but unforgiving.

  1. Confirm that valve is closed (knob should already be tightened clockwise).
  2. Remove the brass cap.
  3. Attach gauge.
  4. Attach brass cap to side of gauge tee fitting.
  5. Open and close valve quickly (a few seconds).
  6. Read vacuum on the gauge.
  7. Record gauge reading on "Initial Vacuum" column of chain-of-custody.
  8. Verify that canister valve is closed and remove gauge.
  9. Replace the brass cap.

- **Leave Residual Vacuum:** A grab sample can be collected either by allowing the canister to reach ambient conditions or by leaving some residual vacuum (e.g., 5 in. Hg) in the canister. In either case, the final vacuum should be noted on the "Final Vacuum" column on the chain-of-custody. This will enable the laboratory to compare the final vacuum with the receipt vacuum (i.e., the vacuum measured upon arrival at the laboratory). If the two readings differ significantly, Client Services will contact you for instructions on how to proceed.

3.1.1 Step-By-Step Procedures for Canister Grab Sampling

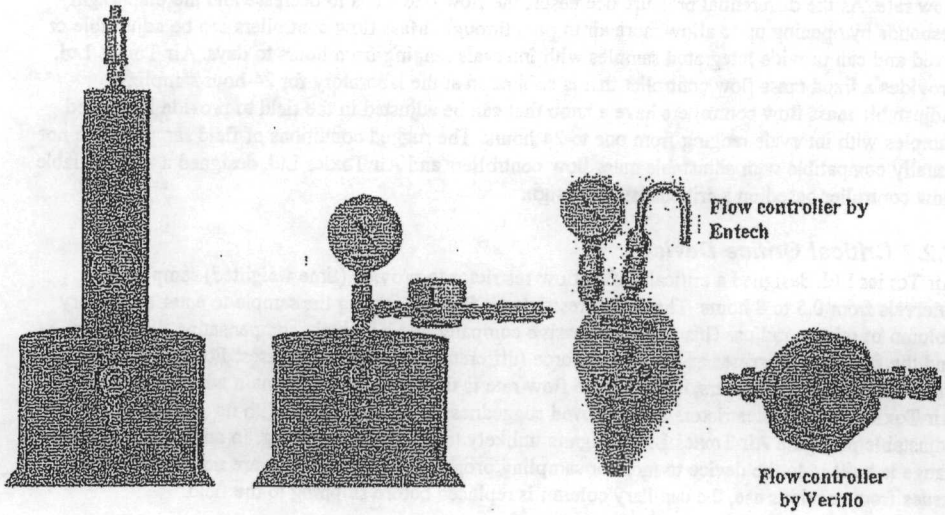
These procedures are for a typical ambient air sampling application and actual field conditions and procedures may vary.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, canister, particulate filter, and gauge – if requested).
2. Verify that gauge is working properly.
3. Verify and record initial vacuum of canister.

When ready to sample:

4. Remove brass cap.
5. Attach particulate filter to canister.
6. Open valve 1/2 turn (6 L canister normally takes about 16 sec to fill).
7. Close valve by hand tightening knob clockwise.
8. Verify and record final vacuum of canister (repeat steps used to verify initial vacuum).
9. Replace brass cap.
10. Fill out canister sample tag.
11. Return canister in box provided
    - Unreturned canister charge of $450 each
12. Return sample media in packaging provided. Unreturned equipment charges:
    - $45 per particulate filter
    - $45 per gauge
13. Fill out chain-of-custody and relinquish samples properly.
14. Place chain-of-custody in box and retain pink copy.

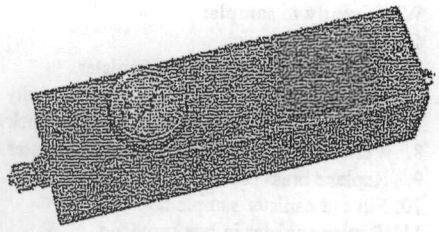


15. Tape box shut and affix custody seal (if applicable) across flap.
16. Ship accordingly to meet method holding times.

3.2 Integrated Sampling with Canisters and Flow Controllers

An air sample collected over more than a few minutes is referred to as an integrated sample and can provide information on compound concentrations in air averaged or composited over time. An 8- or 10-hour integrated sample can be used to determine indoor air quality in the workplace. Similarly, a

24-hour integrated sample can be an economical and practical approach to determine residential exposure to indoor or outdoor air sources. The most common hardware configurations used to take an integrated sample are illustrated.



Flow controllers are devices that regulate the flow of air during sampling into an evacuated canister. Also known as flow restrictors, these devices enable a sampler to achieve a desired flow rate and thus, a sampling interval. Air Toxics Ltd. provides two general types of flow controllers: mass flow controllers and critical orifice devices. Both devices are driven by differential pressure between ambient conditions and vacuum in the canister.

3.2.1 Mass Flow Controller

A mass flow controller employs a diaphragm that actively compensates to maintain a constant mass flow rate. As the differential pressure decreases, the flow rate tends to decrease and the diaphragm responds by opening up to allow more air to pass through. Mass flow controllers can be adjustable or fixed and can provide integrated samples with intervals ranging from hours to days. Air Toxics Ltd. provides a fixed mass flow controller that is calibrated at the laboratory for 24-hour sampling. Adjustable mass flow controllers have a knob that can be adjusted in the field to provide integrated samples with intervals ranging from one to 24 hours. The rugged conditions of field sampling are not usually compatible with adjustable mass flow controllers and Air Toxics Ltd. designed a more reliable flow controller based on a critical orifice design.

3.2.2 Critical Orifice Device

Air Toxics Ltd. designed a critical orifice flow restrictor to provide (time weighted) samples with intervals from 0.5 to 8 hours. The device restricts air flow by forcing the sample to enter a capillary column of minute radius. This device is passive compared to an actively compensating diaphragm and the flow rate decreases as the driving force (differential pressure) decreases. For sampling intervals from 0.5 to 8 hours, however, the flow rate is time weighted. The main advantages of the Air Toxics Ltd. flow restrictors are improved ruggedness and cleanliness. With no moving or adjustable parts, the Air Toxics Ltd. design is unlikely to lose its flow setting. In addition, a vacuum gauge is built in to the device to monitor sampling progress. To ensure there are no contamination issues from previous use, the capillary column is replaced before shipping to the field.

3.2.3 Sampling Interval and Flow Controller Setting

When you request canisters and flow controllers from Air Toxics Ltd., you will be asked for the sampling interval, and the flow controllers will be pre-set prior to shipment according to the table

Table 3.2.3 Flow Rates for Selected Sampling Intervals (mL/min)

Sampling Interval (hrs)	0.5	1	2	4	8	12	24
6 L Canister	167	83.3	41.7	20.8	11.5	7.6	3.5
1 L Canister	26.6	13.3	6.7	-	-	-	-

Note: Target fill volumes for 6 L and 1 L canisters are 5,000 mL and 800 mL, respectively.

below. The flow controller is set to collect 5 L of sample over the sample interval. Final canister vacuum is targeted at 5 in. Hg. The flow rate is set at standard atmospheric conditions (approximately sea level). If the air sample is a process (pressurized or under vacuum) or is collected at elevation, the canister will fill faster or slower depending on sample conditions. If you specify the source at project set-up, we can set the flow controller accordingly. See Section 5.2 for a discussion of collecting a sample at elevation. The 24-hr flow controllers should not be used for process or source samples.

3.2.4 Final Canister Vacuum and Flow Controller Performance

Ideally the final vacuum of a 6 L canister should be 5 in. Hg or greater. As long as the differential pressure is greater than 4 in. Hg ambient pressure, then the flow through the device will remain approximately constant as the canister fills. If there is insufficient differential pressure, the flow through the controller will decrease as the canister pressure approaches ambient. Because of the normal fluctuations in the flow rate (due to changes in ambient temperature, pressure, and diaphragm instabilities) during sampling, the final vacuum will range between 2 and 10 in. Hg.

- **If the residual canister vacuum is greater than 5 in. Hg** (i.e., more vacuum), the flow rate was low and less than 5 L of sample was collected. When the canister is pressurized to 5 psig prior to analysis, sample dilution will be greater than normal. This will result in elevated reporting limits.

- **If the residual canister vacuum is less than 5 in. Hg** (i.e., less vacuum), the initial flow rate

Table 3.2.4 Relationship Between Final Canister Vacuum, Volume Sampled, and Dilution Factor (6 L Canister)

Final Vacuum (in. Hg)	0	2.5	5	7.5	10	12.5	15	17.5	20
Volume Sampled (L)	6	5.5	5	4.5	4	3.5	3	2.5	2
Dilution Factor*	1.34	1.46	1.61	1.79	2.01	2.30	2.68	3.22	4.02

* Canister pressurized to 5 psig for analysis

was high. Once the vacuum decreases below 5 in. Hg, the flow rate begins to drop significantly. This scenario indicates that the sample is skewed in favor of the first portion of the sampling interval.

- **If the final vacuum is near ambient** (i.e., less than 1 in. Hg), there is inadequate differential pressure to drive the flow controller. The sampler cannot be certain the desired sampling interval was achieved before the canister arrived at ambient conditions. The sample could have been acquired over a 1-hour interval (which would be the case if the connection between the canister and flow controller leaked or if the flow controller malfunctioned) or a 24-hour interval. Although the actual sampling interval is uncertain, the canister still contains sample from the site.

3.2.5 Considerations for Integrated Sampling with Canisters

Collecting an integrated air sample is more involved than collecting a grab sample. Sampling considerations include verifying that the sampling train is properly configured, monitoring the integrated sampling progress, and avoiding contamination.

- **Avoid Leaks in the Sampling Train:** See Section 3.1 for instructions on how to securely assemble sampling hardware. A leak in any one of these connections means that some air will be pulled in through the leak and not through the flow controller. A final pressure near ambient is one indication that there may have been a leak.

- **Verify Initial Vacuum of Canister:** See Section 3.1 for instructions on verifying initial canister vacuum. If you are using an Air Toxics Ltd. critical orifice flow controller, note that you can use the built-in gauge. It is important to note both the canister and flow controller serial numbers on the chain-of-custody.

- **Monitor Integrated Sampling Progress:** It is a good idea to monitor the progress of the integrated sampling during the sampling interval. The volume of air sampled is a linear function of canister vacuum. For example, halfway (4 hours) into an 8-hour sampling interval, the canister should be half filled (2.5 L) and the gauge should read approximately 17 in. Hg. More vacuum than 17 in. Hg indicates that the canister is filling too slowly; less than 17 in. Hg and the canister is filling too quickly. If the canister is filling too slowly, a valid sample can still be collected (see Section 3.2.4). If the canister is filling too quickly because of a leak or incorrect flow controller setting, corrective action can be taken. Ensuring all connections are tight may eliminate a leak. It is possible to take an intermittent sample. The time interval need not be continuous.

- **Avoid Contamination:** Flow controllers should be cleaned between uses. This is normally accomplished by returning them to the laboratory. For large air sampling projects, Air Toxics Ltd. has designed a field conditioning program for 24-hour flow controllers involving a purge manifold. This arrangement provides the sampler with scheduling flexibility, inventory control, and convenience in the field. Air Toxics Ltd. will provide the 24-hour flow controllers, a purge manifold, Teflon tubing, rubber ferrules, vacuum pump, and flow meter. The sampler will need to provide the certified nitrogen cylinder and the certified high pressure regulator. Call Client Services at 800-985-5955 if you are interested in the field conditioning program.

- **Keep Sampling Train Out of Direct Sunlight:** The sampling train should be kept out of direct sunlight during sampling. There will be some minor flow rate drift if the temperature of the controllers is allowed to vary significantly.

3.2.6 Step-by-Step Procedures for Integrated Sampling

These procedures are for a typical ambient air sampling application and actual field conditions and procedures may vary.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, canister, particulate filter, and flow controller).
2. Verify initial vacuum of canister.

When ready to sample:

3. Remove brass cap.
4. Attach flow controller to canister.
5. Open valve 1/2 turn.
6. Monitor integrated sampling progress periodically.

At end of sampling interval:

7. Verify and record final vacuum of canister (for 24-hr flow controller repeat steps used to verify initial vacuum and for critical orifice device simply read built-in gauge).
8. Close valve by hand tightening knob clockwise.
9. Replace brass cap.
10. Fill out canister sample tag.
11. Return canisters in boxes provided.
    - Unreturned canister charge of $450 each
12. Return sample media in packaging provided. Unreturned equipment charges:
    - $45 per particulate filter
    - $50-500 per flow controller
13. Fill out chain-of-custody and relinquish samples properly.
14. Place chain-of-custody in box and retain pink copy.
15. Tape box shut and affix custody seal (if applicable) across flap.
16. Ship accordingly to meet method holding times.

Important Information for Canister Sampling

@ DO NOT use canister to collect explosive substances, radiological or biological agents, corrosives, extremely toxic substances, or other hazardous materials. It is illegal to ship such substances and you will be liable for damages.

@ ALWAYS use a filter when sampling. NEVER allow liquids (including water) or corrosive vapors to enter canister.

@ DO NOT attach labels to the surface of the canister.

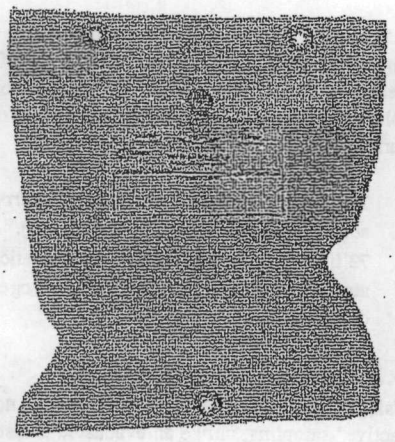
@ DO NOT over tighten the valve and remember to replace the brass cap.

@ IF the canister is returned in unsatisfactory condition, you will be liable for damages.

For help call Client Services at 800-985-5955

Section 4. Sampling with Tedlar Bags

This section provides a description of Tedlar bags, practical considerations for sampling, and step-by-step instructions for collecting a grab sample. Photographs illustrate the correct way to assemble the various sampling components.



4.1 Introduction to Tedlar Bags

A Tedlar bag is a container used to collect a whole air sample for landfill gas, soil gas, and stationary source applications. The Tedlar bag is best suited for projects involving analysis of compounds in the ppmv range. However, Tedlar bags can be used for other applications such as ambient air monitoring for atmospheric/ fixed gases. They can be used to collect sulfur compounds, but only if the fittings are non-metallic (e.g., polypropylene, Teflon, or Nylon).

A Tedlar bag is made of two plies of Tedlar film sealed together at the edges and features a valve that allows the interior to be filled. Sample collection requires a pressurized sampling port, a low flow rate pump, or a lung sampler. The bag expands as sample enters. When the target volume of sample is collected, the valve is closed and the Tedlar bag is returned to the laboratory. Air Toxics Ltd. maintains a limited inventory of Tedlar bags in 1 L , 3 L, and 5 L volumes.

4.1.1 Tedlar Film

Tedlar is a trade name for polyvinyl fluoride film developed by DuPont Corporation in the 1960's. This patented fluoropolymer has been used in a wide variety of applications including protective surfacing for signs, exterior wall panels, and aircraft interiors. Tedlar film is tough, yet flexible and retains its impressive mechanical properties over a wide range of temperatures (well below freezing to over 200° F). Tedlar exhibits low permeability to gases, good chemical inertness, good weathering resistance, and low off-gassing.

4.1.2 How "Active" is the Surface of a Tedlar Bag?

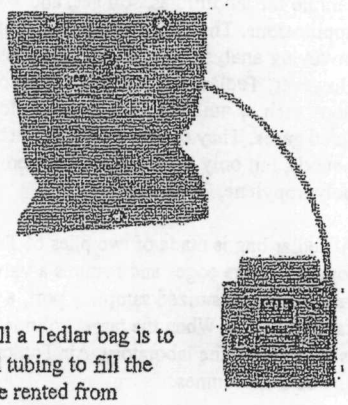
The surface of a Tedlar bag is a work in progress. The surface of a new bag is essentially free of VOCs at the single digit ppbv level. Compounds detected from analyzing new Tedlar bags include methylene chloride, toluene, acetone, ethanol; and 2-propanol. Note that 2-propanol has been detected in some new bags up to 45 ppbv. Once the Tedlar bag is used, however, the surface has been exposed to moisture and possibly VOCs. It may irreversibly adsorb many VOCs at the low ppbv level. A series of purges with certified gas may not remove the VOCs from the surface. $15 for a new bag is a small price to pay for peace of mind.

⇨ **Never reuse a Tedlar bag when sampling for ppbv level compounds.**

4.1.3 Hold Time for a Tedlar Bag

The media hold time for a Tedlar bag is indefinite if stored out of sunlight in a cool, dry location. Tedlar bags can be used to collect samples containing common solvents, hydrocarbons, chlorinated solvents, sulfur compounds, and many other classes of compounds. The sample hold time to analysis varies for different classes of compounds:

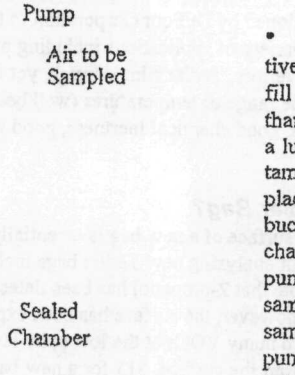
- **24 hours:** Sulfur compounds (e.g., hydrogen sulfide and methyl mercaptan) and chemically active compounds (e.g., 1,3-butadiene).
- **72 hours:** Chlorinated solvents, aromatic compounds, and atmospheric/fixed gases (oxygen, nitrogen, carbon dioxide).



4.2 Tedlar Bag Sampling

Using a Tedlar bag to collect an air sample normally involves "active" sampling, unlike an evacuated canister that can be filled "passively" by simply opening the valve. There are two methods commonly used to fill a Tedlar bag: using a pump or a lung sampler.

- ***Sampling with a Pump***: The most common method to fill a Tedlar bag is to use a small pump with low flow rates (50-200 mL/min) and tubing to fill the bag. Air Toxics Ltd. does not provide pumps but they can be rented from equipment providers or purchased from manufacturers such as Neuberger or Gilian.



- ***Sampling with a Lung Sampler***: Alternatively to using a pump, a "lung sampler" can be used to fill a Tedlar bag. Although a little more complicated than simply using a pump, the main advantage to using a lung sampler is that it avoids potential pump contamination. A Tedlar bag with attached tubing is placed in a small airtight chamber (even a 5-gallon bucket can work) with the tubing protruding from the chamber. The sealed chamber is then evacuated with a pump causing the bag to expand and drawing the sample through the protruding tube into the bag. The sample air never touches the wetted surfaces of the pump. Air Toxics Ltd. does not provide lung samplers, but they can be rented from equipment suppliers or

purchased by manufacturers such as SKC Inc.

4.2.1 Considerations for Tedlar Bag Sampling

The following are some considerations for collecting a Tedlar bag sample.

- **Fill the Tedlar bag no more than 2/3 full:** Allow for possible expansion due to an increase in temperature or decrease in atmospheric pressure (e.g., the cargo hold of a plane).

- **Keep the Tedlar bag out of sunlight:** Tedlar film is transparent to ultraviolet light (although opaque versions are available) and the sample should be kept out of sunlight to avoid any photochemical reactions.

- **Protect the Tedlar bag:** Store and ship the Tedlar bag samples in a protective box at room temperature. An ice chest can be used, but DO NOT CHILL.

- **Fill out the Tedlar bag label:** It is much easier to write the sample information on the label before the Tedlar bag is inflated.

- **Provide a second Tedlar bag:** Consider filling two bags per location in the rare occasion that a defective bag deflates before analysis.

- **Avoid Contamination:** Care should be taken to avoid contamination introduced by the pump or tubing. Begin sampling at locations with the lowest compound concentrations (e.g., sample the SVE effluent before the influent). Decontaminate the pump between uses by purging with certified air for an extended period; better yet, use a lung sampler. Use shortest length possible of Teflon tubing or other inert tubing. Do not reuse tubing. If long lengths of tubing are used, consider purging the tubing with several volumes worth before sampling. If you are concerned about sampling for trace compounds, you shouldn't be using a Tedlar bag (see Section 1.2).

- **Don't Sample Dangerous Compounds in a Tedlar Bag:** Do not ship any explosive substances, radiological or biological agents, corrosives, or extremely hazardous materials to Air Toxics Ltd.

Tedlar bag rupture during transit to the laboratory is possible and the sampler assumes full liability.

4.2.2 Step-by-Step Procedures for Tedlar Bag Sampling (Pump)

Note: These procedures are for a typical stationary source (e.g., SVE system) sampling application; actual field conditions and procedures may vary. See additional sampling considerations in Section 5.3 for sampling soil gas or landfill gas.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, Tedlar bag, and tubing/fittings – if requested).
2. Verify pump cleanliness and operation (Air Toxics Ltd. does not provide pumps).

When ready to sample:

3. Purge sample port.
4. Attach new Teflon tubing from sample port or probe to low flow rate pump.
5. Purge tubing.
6. Fill out Tedlar bag sample tag.
7. Attach additional new Teflon tubing from the pump outlet to the Tedlar bag valve.
8. Open Tedlar bag valve.
9. Collect sample (FILL NO MORE THAN 2/3 FULL).
10. Close Tedlar bag valve by hand tightening valve clockwise.
11. Return Tedlar bag in boxes provided (DO NOT CHILL).
12. Fill out chain-of-custody and relinquish samples properly.
13. Place chain-of-custody in box and retain pink copy.
14. Tape box shut and affix custody seal (if applicable) across flap.
15. Ship priority overnight to meet method holding times. 3 DAY HOLD TIME TO ANALYSIS (most analyses).

Section 5. Special Sampling Considerations

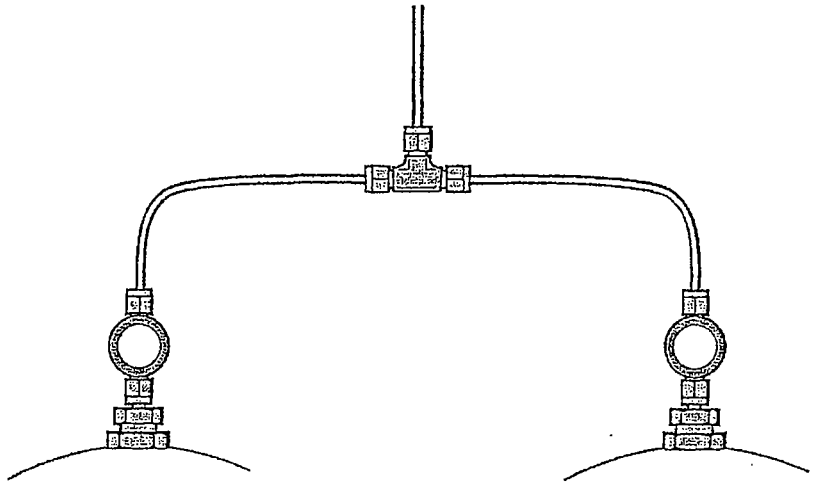
This section provides considerations for special sampling configurations that a sampler may collect in the field such as a field duplicates or an ambient blank. This section also provides considerations for sampling at altitude, soil/landfill gas sampling, and sample cylinder sampling.

5.1 Special Sampling Configurations

Special sampling configurations include a field duplicate, field split, field blank, ambient blank, trip blank, and an equipment rinse. Call Client Services at 800-985-5955 if your project involves any of these special sampling configurations.

5.1.1 Field Duplicate

A field duplicate is a second sample collected in the field simultaneously with the primary sample at one sampling location. The results of the duplicate sample can be compared (e.g., calculate relative percent difference) with the primary sample to provide information on consistency and reproducibility of field sampling procedures. Due to the nature of the gas phase, duplicate samples should be collected from a common inlet. The configuration for collecting a field duplicate includes stainless steel or Teflon tubing connected to a Swagelok "tee".



5.1.2 Field Blank

A field blank is a sample collected in the field from a certified air source. Analysis of the field blank can provide information on the decontamination procedures used in the field. Clean stainless steel or Teflon tubing and a certified regulator should be used. It is imperative that individually certified canisters (the sample canister and the source canister/cylinder, if applicable) be used to collect a field blank.

5.1.3 Ambient Blank

An ambient blank is an ambient air grab sample collected in the field normally used in conjunction with soil gas or stationary source (e.g., SVE system) sampling. Analysis of the ambient blank can provide information on the ambient levels of site contaminants. It is imperative that an individually certified canister be used to collect an ambient blank.

5.1.4 Trip Blank

When sampling for contaminants in water, the laboratory prepares a trip blank by filling a VOA vial with clean, de-ionized water. The trip blank is sent to the field in a cooler with new sample vials. After sampling, the filled sample vials are placed back in the cooler next to the trip blank and returned to the laboratory. Analysis of the trip blank provides information on decontamination and sample handling procedures in the field as well as the cleanliness of the cooler and packaging.

When sampling for compounds in air, a trip blank provides little, if any, of the information above. A trip blank canister can be individually certified, evacuated, and sent to the field in a box with the sample canisters. Since the valve is closed and the brass cap tightened, it is questionable if the trip blank canister contents are ever "exposed" to sampling conditions. At the laboratory, the trip blank canister will be pressurized prior to analysis with dry, nitrogen – a matrix that may be entirely different than the sampled air. The recovery of target compounds can vary by matrix (e.g., moisture, carbon dioxide) rendering the trip blank results meaningless. **Air Toxics Ltd. does not recommend analyzing a trip blank for air sampling.**

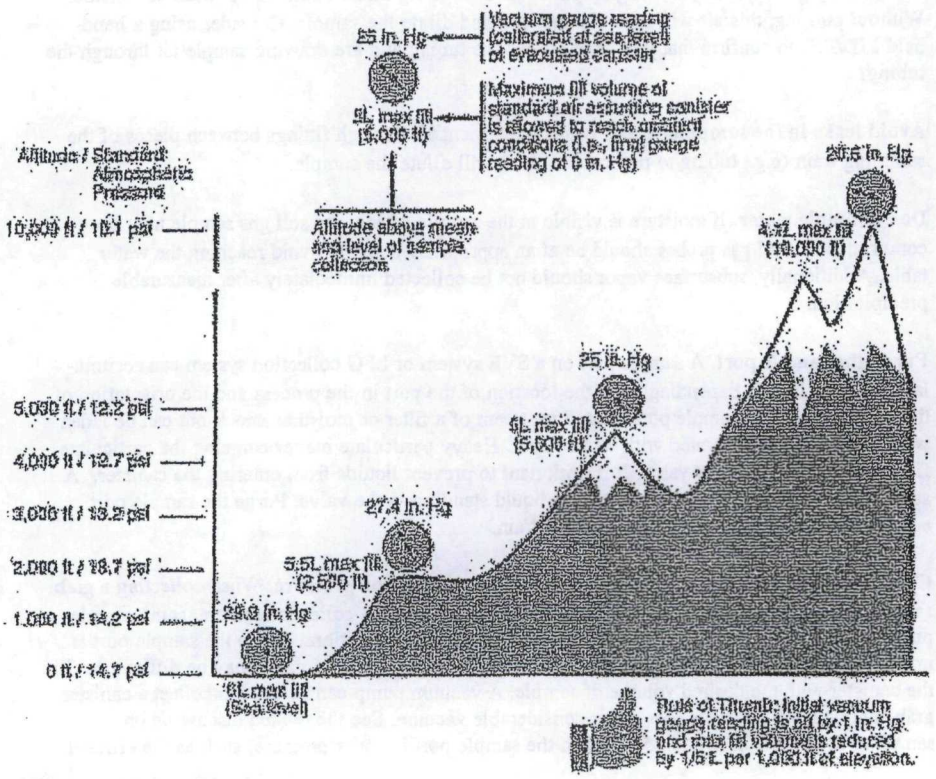
5.2 Considerations for Sampling at Altitude

Sampling at altitudes significantly above sea level is similar to sampling a stationary source under vacuum in that target fill volumes may be difficult to achieve. The figure below illustrates the relationship between increasing altitude and decreasing atmospheric pressure. Ambient conditions in Denver at *5,000 ft altitude are quite different than ambient conditions at sea level. Canister sampling* is driven by the differential pressure between ambient conditions and the vacuum in the canister. There is less atmospheric pressure in Denver and 5 L is the maximum fill volume of standard air assuming the canister is allowed to reach ambient conditions (i.e., final gauge reading of 0 in. Hg). Theoretically, if you sample high enough (e.g., in space), no sample would enter the canister because there is no pressure difference between the evacuated canister and ambient conditions. To fill a canister to 6 L in Denver, you would need to use an air pump.

Sampling at altitude also affects gauge readings. The gauges supplied by Air Toxics Ltd. (see Section 2.2.4) measure canister vacuum relative to atmospheric pressure and are calibrated at approximately sea level. Before sampling at altitude, the gauges should be equilibrated (see Section 3.1). But even after equilibrating the gauge, verifying the initial vacuum of a canister at altitude is misleading. In Denver at 5,000 ft, expect the gauge to read 25, not 29.9 in. Hg. You do not have a bad canister (i.e., leaking or not evacuated properly). The canister is ready for sampling and the gauge is working properly.

⇨ Rule of Thumb: For every 1,000 ft of elevation, the gauge will be off by 1 in. Hg and the fill volume will be reduced by 1/5 L.

If you have questions about sampling at altitude, please call Client Services at 800-985-5955.



5.3 Considerations for Soil/Landfill Gas Sampling

There are some additional sampling considerations for collecting grab samples (canister or Tedlar bag) from a soil boring, landfill boring, SVE system, or landfill gas (LFG) collection system. The general challenge with these samples arises from the need to employ long lengths of tubing to direct the soil gas, landfill gas, or process air to the canister or Tedlar bag. Tubing introduces the potential for contamination and diluting the sample.

- **Use inert tubing.** Teflon tubing is recommended. Tubing with an outer diameter of 1/4 in. works best with the fittings on the particulate filter.

- **Do not reuse tubing.** $2 per foot for new tubing is a small price to pay for peace of mind.

- **Purge tubing adequately.** A long length of tubing has significant volume of "dead air" inside. Without purging, this air will enter the canister and dilute the sample. Consider using a hand-held PID/FID to confirm that you have purged the tubing and are drawing sample air through the tubing.

- **Avoid leaks in the sampling train.** Leaks of ambient air through fittings between pieces of the sampling train (e.g., tubing to particulate filter) will dilute the sample.

- **Don't sample water.** If moisture is visible in the sample tubing, the soil gas sample may be compromised. Soil gas probes should be at an appropriate depth to avoid reaching the water table. Additionally, subsurface vapor should not be collected immediately after measurable precipitation.

- **Purge the sample port.** A sample port on a SVE system or LFG collection system can accumulate solids or liquids depending upon the location of the port in the process and the orientation of the port. An influent sample port located upstream of a filter or moisture knock-out can be laden with particulates or saturated with water vapor. Heavy particulate matter can clog the particulate filter and foul the canister valve. It is important to prevent liquids from entering the canister. A sample port oriented downward may have liquid standing in the valve. Purge the sample port adequately before connecting the sampling train.

- **Consider the effects of sampling a process under vacuum or pressure.** When collecting a grab sample from a stationary source such as an SVE system or LFG collection system, some sample ports may be under vacuum or pressure relative to ambient conditions. When the sample port is under vacuum, such as the header pipe from the extraction well network, it may be difficult to fill the canister with the desired volume of sample. A vacuum pump can be used to collect a canister grab sample from a sample port under considerable vacuum. See the related discussion on sampling at altitude in Section 5.2. When the sample port is under pressure, such as the effluent

stack downstream of the blower and treatment system, you may inadvertently pressurize the canister. Only a DOT-approved sample cylinder should be used to transport pressurized air samples (see Section 5.4). Under no circumstances should an Air Toxics Ltd. canister be pressurized more than 5 psig for a 6 L canister and 15 psig for a 1 L canister. Bleed off excess pressure by opening the valve temporarily while monitoring the canister with a pressure gauge.

5.4 Considerations for Sample Cylinder Sampling

Sample cylinders, also known as "sample bombs", are DOT-approved, high pressure, thick-walled, stainless steel cylinders with a valve at each end. They were intended for collecting a pressurized sample for petroleum gas applications. Sample cylinders differ from sample canisters in that they do not have a Summa-passivated interior surface and are not evacuated prior to shipment. Sample cylinders are not suitable for analysis of hydrocarbons at ppbv levels. Sample cylinders can be used for analysis of natural gas by ASTM D-1945 and calculation of Btu by ASTM D-3588. Air Toxics Ltd. assumes that clients requesting a sample cylinder have a pressurized process and sample port with a built-in gauge and 1/4 in. Swagelock fitting to attach to the sample cylinder. Air Toxics Ltd. has an inventory of 500 mL sample cylinders that are particularly suited for landfill gas collection systems (i.e., LFG to energy applications). This section provides step-by-step procedures for sampling with a sample cylinder.

Step-by-Step Procedures for Sample Cylinder Sampling

These procedures are for a typical stationary source sampling application and actual field conditions and procedures may vary. Follow all precautions in the site Health and Safety Plan when dealing with a pressurized sample port and sample cylinder.

1. Verify contents of the shipped package (e.g., chain-of-custody, sample cylinder, particulate filter).
2. Verify that gauge on sample port is working properly.
3. Purge sample port.
4. Remove brass caps on either end of cylinder.
5. Attach particulate filter to upstream valve.
6. Attach filter/cylinder assembly directly to the sample port.
7. Open both valves 1/2 turn.
8. Allow sample air to flow through sample cylinder (approximately 10 L for a 500 mL cylinder).

A P P E N D I X C-6

Soil Waste Characterization Sampling Procedures

ATTACHMENT C-6

Soil Waste Characterization Sampling Procedures

A. Introduction

Soil waste sampling activities will be performed during construction of the augmented soil vapor extraction (SVE) system. The procedures to be used during the collection of soil waste characterization samples from stockpiles of excess soils excavated from the augmented SVE trench segments are described below.

B. Soil Sample Collection Procedures

1. Field Screening

Soils at five locations across each soil stockpile will be field screened for volatile organic compounds (VOCs) using a portable photoionization detector (PID) with an 11.8 eV bulb. An approximately 1-foot deep hole will be dug at each location using a shovel. A PID measurement will be made immediately, placing the probe near the base of the hole for approximately 15 seconds until the instrument has measured a maximum reading.

2. Soil Sampling

At least one sample will be collected from the soil stockpile for each augmented SVE trench segment for laboratory analysis (i.e., a total of 7 samples), at the locations that exhibit the highest PID instrument response. The procedure for collecting waste soil samples (except non-SPLP VOC analysis samples) will be as follows:

- At least 1 foot of soil will be scraped from the surface of the pile. The samples will be collected from the newly exposed pile surface and placed in the sample jars using decontaminated sample spoons or scoops.
- Samples will be collected by directly filling the sample containers with the appropriate sample volumes.

- Sample containers will be labeled with the sample ID, time, date, sampler and analysis.
- Samples will be placed on ice and ship to CompuChem Laboratories within the holding time.

Soil samples for VOCs (non-leachate analyses only) will be collected using United States Environmental Protection Agency (USEPA) SW-846 Method 5035 (methanol field preservation method or equivalent methods). The procedure for collection waste soil samples for VOC analysis will be as follows:

- Prepare sampling area of the pile as described above.
- Push a laboratory-supplied sample syringe into the pile surface to fill the syringe to the 5 gram mark.
- Extrude the 5-gram soil sample directly into a laboratory-prepared glass (40-ml) vials and replace the cover as soon as possible.
- Repeat, collecting a 5-gram sample for each of the three laboratory-supplied vials (two containing sodium bisulfate preservative and one containing methanol preservative).
- Fill a 4-ounce jar with soils from the same area of the VOC samples for moisture content analysis.
- Label each vial and jar with sample identifier, time, date, sampler and analysis.
- Place samples on ice and ship to CompuChem Laboratories within 24 hours.

APPENDIX C-7

Decontamination Procedures

Table 6-1. Decontamination Protocol for Sampling Equipment

Step Number	Description
1	Scrub equipment thoroughly with soft-bristled brushed in a low-suds detergent solution.
2	Rinse equipment with tap water by submerging and/or spraying.
~~3~~	~~Rinse equipment with methanol by spraying until dripping; retain drippings.~~
4	Rinse equipment with distilled water by spraying until dripping; retail drippings.
5	Rinse equipment with distilled water a second time by spraying until dripping; retain drippings.
6	Place equipment on plastic or aluminum foil and allow to air dry for 5 to 10 minutes.
7	Wrap equipment in aluminum foil (shiny side out) for handling and/or storage until next use.

6.3.1 Water Level Measurement

Static water levels will be measured to the nearest 0.01 foot in each monitoring well and the piezometer at each sampling event and recorded in the field notebook. The water level surface will be measured prior to well purging and sampling by using an electric water level meter. Before lowering the probe in the well, the batteries will be checked by pressing the test button on the instrument. The probe will be slowly lowered into the well until contact with the water surface is indicated on the meter. The probe will be withdrawn just above the water surface, and a second reading will be taken prior to withdrawing the probe from the well. Both readings will be recorded in the field logbook. The probe will be decontaminated prior to inserting the instrument into a well by washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water.

Each well will have a reference point, indicated on the inner well casing, from which water level measurements will be taken. The reference point elevation on the well will be

APPENDIX C-8

Sample Handling and Analysis Procedures

7.0 Sample Handling and Analysis

The required sample containers, preservation methods, maximum holding times, and filling instructions for each sample type are summarized on Table ~~7-1~~ E-6.

Sample bottles provided by the selected analytical laboratory will be prepared by using the procedures required by the Contract Laboratory Program (CLP). Sample bottles provided by the selected analytical laboratory will be prepared as described in their standard operating procedure (SOP). ~~Sample tubes for extracted soil vapor will also be provided by the selected analytical laboratory. Reference to sample chain-of-custody procedures are contained in Volume I, Section 5.0.~~

Waste generated onsite will be properly handled and disposed of to prevent contamination of clean areas in accordance with ~~Technical Specification 02080 Remedial Action Generated Wastes.~~ the Design Report.

7.1 Sample Packaging and Shipment

Following sampling, the outside of the sample bottles will be rinsed with potable or distilled water near the sampling location. The sample packaging and shipment procedures will be as follows:

- The sample will be properly preserved (if applicable) and liquid levels will be marked if bottles are partially full;
- Custody tags will be securely attached to the sample container, and each container will be placed in a Ziploc bag;
- The sample containers will be placed in a cooler lined with 2 inches of vermiculite or equivalent absorbent material and maintained at 4°C with cold packs or ice sealed in plastic bags as appropriate. The remaining space in the cooler will be filled with additional packing material;

Table 7-1. Sample Containers, Preservatives, and Holding Times

Analysis	Container Type	Preservation and Storage Requirements	Maximum Holding Time
Soil-VOC	Two 8-ounce glass jars[a]	4°C; protect from light	14 days
Soil-1,2-Dichlorobenzene Phenol	Two 8-ounce glass jars[a]	4°C; protect from light	14/40 days[b]
Water-VOCs	Three 40-mL glass vials[a]	HCl to pH≤ 2; 4°C; protect from light	14 days
Water-BNAs	Two 1-liter amber glass jars[a]	4°C; protect from light	7/40 days[b]
Water-PCBs	Two 1-liter amber glass jars[a]	4°C; protect from light	7/40 days[b]
Water-Metals	One 1-liter poly bottle[a]	HNO_3 to pH≤ 2; 4°C; protect from light	6 months (Mercury = 28 days)
Water-Chromium VI (CR +6)	One 1-liter poly bottle[a]	4°C; protect from light	24 hours
Water-Alkalinity	One 250-mL poly bottle[a]	4°C	14 days
Water-TDS	One 250-mL poly bottle[a]	4°C	7 days
Water-TSS	One 250-mL poly bottle[a]	4°C	7 days
Water-Cyanide	One 500-mL poly bottle[a]	NaOH to pH>4°C	14 days

[a]Teflon-lined cap or septa
[b]Days to extraction/days of analysis

- The cooler will be closed and sealed shut with strapping tape. If the cooler has a drain port, that port will also be sealed shut with tape. One custody seal will be placed across the front of the cooler, and another seal will be affixed across the hinge area at the back of the cooler. These custody seals will be covered with clear tape;
- An airbill with shipper's and consignee's addresses will be affixed to the top of the cooler. If liquid samples are being shipped, "This End Up" labels will be placed appropriately.
- The samples will be shipped to the appropriate laboratory by using an overnight service; and
- The laboratory will be notified that it will be receiving the samples.

~~Sample custody procedures are detailed in Volume I, Section 5.0. The samples to be analyzed for chromium VI will be hand delivered to the selected analytical laboratory.~~

7.2 Sample Analysis

Samples will be analyzed following the methods listed in the QAPP. ~~Volume I, Section 7.0.~~

APPENDIX D

Design Parameters for Permeable Reactive Gate System

APPENDIX D

Design Parameters for Permeable Reactive Gate System

The Permeable Reactive Gate System (PRGS) is being installed to treat any subsurface water at the Enviro-Chem Superfund Site ("ECC Site" or the "Site") that infiltrates into the augmented SVE trenches and increases the water level in the trenches to the elevation of the perforated collection pipe. At that point, the water will flow out of the trenches, through underground drainage pipes, to the PRGS collection manhole and then pumped to the PRGS treatment vessel. Cis-1,2-dichloroethene (cis-1,2-DCE) is the constituent of concern currently present in the till water requiring the longest detention time based on published information on degradation rates and maximum concentrations historically detected in surface and subsurface water samples. As such, the PRGS is designed to treat cis-1,2-DCE to below the corresponding Effluent Limit for Discharge of Treated Water to Unnamed Ditch of 2.0 ug/L (Design Report, Table 2-3).

Design Flow Rate

The design flow rate for the PRGS is based on the anticipated discharge of till water into the augmented SVE trenches and subsequent flow of the water through the collection piping to the PRGS treatment vessel. Two design flow rates were calculated for the PRGS: one is the average design flow rate that represents flow under typical site conditions and the second is the maximum design flow rate that represents flow under high intensity rainfall conditions (i.e., a significant magnitude and short duration precipitation event). The calculation of the average design flow assumes the flow into the PRGS is equivalent to the flow through the portion of the shallow till unit that is intersected by the thin barrier curtain wall (TBCW). A form of Darcy's Law was used to calculate the average design flow rate (Q_A) for the PRGS.

$$Q_A = KAI$$

Where:

K = hydraulic conductivity (feet per day [ft/d]).

A = saturated cross-sectional area perpendicular to the till water flow direction (square feet [ft^2]).

I = hydraulic gradient (feet per foot [ft/ft])

The hydraulic conductivity of the till unit, 0.0251 ft/d, is from the 2006 till water extraction testing at well HS-2 reported in ENVIRON's August 2006 *Till Water Pump Testing Report* (the "Pump Testing Report"). The saturated cross-sectional area perpendicular to the till water flow

direction is equivalent to the area defined by the transect extending from the north end of augmented SVE trench segment 4 to the north end of augmented SVE Trench Segment 7, as shown on Drawing C-3 (i.e., 294 feet), and a conservative estimate of the saturated depth of the barrier wall near the north end of augmented SVE Trench Segment 4 (25 feet). The hydraulic gradient is the maximum historical gradient between T-10 and T-8 calculated from the water level measurements during the 1999 through 2002 till water sampling events.

The calculations for the average design flow rate are as follows:

K = 0.0251 ft/d
A = (294 ft * 25 ft) or 7,350 ft^2
I = 0.011 ft/ft
Q_A = 2.03 ft^3/d or 15.2 gallons per day (gpd)

Based on these calculations, the average design flow rate (Q_A) is expected to be approximately 15.2 gpd.

The maximum design flow rate (Q_P) assumes periodic intense precipitation events may cause water levels within the augmented SVE trench segments to rise significantly over short periods of time. Although the design of the augmented SVE trench segments (covering the trench segments with geomembrane and compacted fill) minimizes the likelihood of such rises, for design purposes, it is assumed that water levels may rise as much as 0.25 feet during a significant rain event (based on a rise in water level of less than approximately 0.25 feet measured in wells HS-1, HS-1A, PS-2, and PT-3 during a 1.6 inch precipitation event that lasted less than one hour during the July 2006 till water extraction testing).

The design dimensions of the augmented SVE trenches are shown on Drawing C-3 (width of 2 feet and combined length of 985 feet). A uniform 0.25-foot rise in water levels within the augmented SVE trenches and a porosity (n) of the augmented SVE trench backfill of 0.30 are assumed. Based on these assumptions, the additional volume of water within the trenches is expected to be approximately 1,105 gallons.

The calculations for the peak design flow rate are as follows:

V_W = Saturated Volume (V_S) x Porosity or $V_W = V_S n$
V_S = 985 ft x 2 ft x 0.25 ft or 492.5 ft^3
n = 0.30
V_W = 147.75 ft^3 or 1,105 gallons

Assuming this additional volume of water is discharged from the augmented SVE trenches over a 24-hour period:

$$Q_P = V_W / t$$

Based on these calculations, the maximum design flow rate (Q_P) is expected to be approximately 1,105 gpd.

Residence Time and Volume of Granular Iron

Based on a data base maintained by EnviroMetal Technologies, Inc. (ETI), a residence time of approximately 41 hours (1.7 days) would be required to reduce the highest detected concentration of cis-1,2-DCE (110 ug/L; in till well T-9, located in the vicinity of Trench Segments 5, 6, and 7) to below the corresponding Effluent Limit for Discharge of Treated Water to Unnamed Ditch of 2.0 ug/L.[1] The volume of granular iron required in the PRGS reactor vessel is calculated as follows:

$$V_I = Qt/n$$

Where:

Q = design flow rate (147.75 ft^3/day)

t = residence time (1.7 days)

n = porosity (0.4 for 100% granular iron)

V_I = 627 ft^3 or 47 tons (using a bulk density of for the granular iron of 0.075 tons/ ft^3, as provided by ETI)

Based on these calculations, the amount of granular iron needed within the PRGS reactor vessel is approximately 627 ft^3 or 47 tons.

PRGS Reactor Tank Dimensions and Head Requirements

Based on design details provided by ETI, the reactor vessel is divided into three separate sections: a pretreatment zone; a 100% iron zone; and an effluent zone (Drawing C-12). Flow occurs downwards through the pretreatment and iron zones. The interior dimensions of the PRGS reactor vessel needed to contain the required amount of granular iron are as follows:

- Pretreatment zone – length of 2 ft, width of 13 ft, and depth of 4 ft;
- 100% iron zone – length of 13 ft, width of 13 ft, and depth of 4 ft;

[1] Calculated using a degradation rate of 0.099 hr^{-1} provided by ETI based on first-order degradation simulations.

Therefore, the head difference needed to promote vertical flow in the pretreatment zone is calculated as follows:

Cross-sectional area (A) = 2 ft x 13ft = 26 ft^2
Specific Discharge (v) = Q/A = 148 ft^3/day/26 ft^2 = 5.7 ft/day
Δz = 4 ft, K = 50 ft/day (for a iron/sand mix)
Δh = v x Δz / K = 0.5 ft

Similarly, the head difference needed to promote vertical flow in the 100% iron zone is calculated as follows:

Cross-sectional area (A) = 13 ft x 13 ft = 169 ft2
Specific Discharge (v) = Q/A = 148 ft3/day/169 ft2 = 0.87 ft/day
Δz = 4 ft, K = 70.9 ft/day (for 100% granular iron)
Δh = v x Δz / K = 0.05 ft

Based on these calculations, the total head difference required to promote flow through the reactor vessel is less than 1 foot.

APPENDIX E

Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN
ATTACHMENT Z-1 REMEDY

ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Submitted to

United States Environmental Protection Agency, Region 5

Submitted by

ENVIRON International Corporation
Deerfield, Illinois

On behalf of

Enviro-Chem Site Trust Fund

September 2006
Revised February 2007

USEPA Region 5 Remedial Project Manager	Date
USEPA Region 5 Quality Assurance Reviewer	Date
IDEM Project Manager	Date
Project Coordinator for Non-Premium Respondents	Date
ENVIRON QA Director	Date
CompuChem Laboratory QA Manager	Date
Air Toxics Laboratory Director	Date
General Contractor Project Manager	Date

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(continued)

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(continued)

TABLES

FIGURES

ATTACHMENTS

ACRONYMS/ABBREVIATIONS

°C	degrees Celsius
%RPD	relative percent difference
%R	percent recovery
CRQL	Contract Required Quanitation Limits
DQO	Data Quality Objective
ECC	Enviro-Chem
ECC Site	Enviro-Chem Superfund Site
ENVIRON	ENVIRON International Corporation
ERM-North Central	Environmental Resources Management-North Central, Inc.
FSP	Field Sampling Plan
GC/MS	gas chromatography/mass chromatography
IDEM	Indiana Department of Environmental Management
ILs	instrument detection limits
MDLs	method detection limits
MS	matrix spike
MSD	matrix spike duplicate
MS/MSD	matrix spike/matrix spike duplicate
NFGI	National Functional Guidelines for Inorganic
NFGO	National Functional Guidelines for Organic
O&M	operations and maintenance
OSHA	Occupational Safety and Health Act
PID	photoionization detector
ppb	parts per billion
PRGS	permeable reactive gate system
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RLs	reporting limits
ROD	Record of Decision
Site	Enviro-Chem Superfund Site
SOPs	Standard Operating Procedures
SVE	soil vapor extraction
SVOC	semi-volatile organic compound
SW-846	Test Methods for Evaluating Solid Waste
TBCW	thin barrier curtain wall
TSA	Technical System Audit
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

I. GROUP A: PROJECT MANAGEMENT

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) activities associated with the augmentation of the soil vapor extraction (SVE) system at the former Enviro-Chem (ECC) Superfund Site located in Zionsville, Indiana (the "ECC Site" or the "Site"). This QAPP also describes the specific protocol that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis. All QA/QC procedures will be conducted in accordance with applicable professional technical standards, United States Environmental Protection Agency (USEPA) requirements and guidelines, and specific project goals and requirements.

This QAPP has been prepared in accordance with USEPA guidance documents, specifically, *EPA Requirements for Quality Assurance Project Plans, EPAQA/R-5 Interim Final* (USEPA 1999) and *Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Region 5* (USEPA 2000). This document is provided as Appendix E to the *Design Report for the Augmentation of SVE System* (the "Design Report").

A. Distribution List

At a minimum, the following individuals will be provided with copies of the QAPP:

- USEPA Region 5 Project Manager – Matthew Ohl;
- Indiana Department of Environmental Management (IDEM) Project Manager – Bruce Hamilton;
- Project Coordinator for Non-Premium Respondents – Ronald E. Hutchens, P.E. (ENVIRON International Corporation [ENVIRON]);
- ENVIRON Field Activities Coordinator – Cynthia Bonczkiewicz, P.E.;
- ENVIRON QA Director – Felix Moran, P.E.;
- CompuChem Project Manager – Marlene Smith;
- CompuChem QA Officer – Valgena Respass;
- Air Toxics Project Manager – Brandon Dunmore;
- Air Toxics QA Officer – Melanie Levesque;
- MAKuehl Company – Marcia Kuehl; and
- Contractor (to be inserted following selection by the ECC Trust and approval by USEPA).

B. Project Organization

On behalf of the ECC Site Trust Fund (the "Trust"), the Contractor or the Trust-appointed operations manger will have overall responsibility for the field activities and the quality of the work. On behalf of the Trust, ENVIRON will provide on-site engineering oversight, project coordination, and implementation of certain sampling/monitoring activities associated with the Attachment Z-1 Remedy. Fixed-base laboratory analyses for the soil and water samples will be performed by CompuChem Laboratories. Laboratory analysis of soil vapor samples will be performed by Air Toxics. Independent data validation will be performed by MAKuehl Company. The various QA and management responsibilities of key project personnel are defined below. A generalized Project Organization Chart is included as Figure E-1.

1. Management Responsibilities

- USEPA Project Manager – The USEPA Remedial Project Manager, Matthew Ohl, has the overall responsibility for USEPA regulatory oversight of all phases of the Attachment Z-1 Remedy. The USEPA Remedial Project Manager will be responsible for reviewing and approving the QAPP.

- IDEM Project Manager – The IDEM Project Manager, Bruce Hamilton, has the overall responsibility for IDEM regulatory oversight of all phases of the Attachment Z-1 Remedy.

- Project Coordinator for Non-Premium Respondents – Ronald E. Hutchens, P.E., ENVIRON, has overall responsibility for ensuring the quality of work described in the Design Report, on behalf of the ECC Trust. Mr. Hutchens will have overall responsibility for:

  - Establishing project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task in cooperation with the Trust;

  - Developing and meeting ongoing project and/or task staffing requirements for the Trust's monitoring/sampling activities, including mechanisms to review and evaluate each task product;

- Reviewing the work performed by the Contractor to ensure its quality, responsiveness, and timeliness;

- Approving all external reports (deliverables) before their submission to USEPA Region 5 and IDEM;

- Ensuring the preparation and quality of monthly progress reports and draft and final Design Reports;

- Orienting the Contractor, field staff, and support staff concerning the project's special considerations; and

- Monitoring and directing ENVIRON's design and monitoring teams.

- Project Manager for Contractor – (Name and company to be inserted following selection by the Trust and approval by USEPA). The Project Manager for the Contractor has overall responsibility for ensuring the quality of construction and operations and maintenance (O&M) work performed on behalf of the Trust. Some sampling/analytical activities will be the responsibility of the Contractor also. O&M work may be subcontracted by the Contractor or contracted directly with the Trust.

2. Quality Assurance Responsibilities

- USEPA Quality Assurance Reviewer – The USEPA will designate a reviewer for this QAPP upon submittal. The reviewer will check the QAPP for compliance with current EPA requirements and provide comments regarding any differences or approval for use of the document for the subject project.

- ENVIRON QA Director – The ENVIRON QA Director is Felix Moran, P.E. The QA Director will remain independent of direct job involvement and day-to-day operations, and have direct access to project staff, as necessary, to resolve any QA dispute. The QA Director will be responsible for auditing the implementation of the QA program in conformance with the demands of

specific investigations, ENVIRON's policies, and USEPA/IDEM requirements. Specific functions and duties include:

- Reviewing and approving QA plans and procedures;
- Performing QA audits on various phases of the field operations;
- Providing QA technical assistance to project staff; and
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the ENVIRON Project Coordinator.

- CompuChem and Air Toxics Laboratory QA Officers – The Laboratory QA Officers will:

  - Oversee laboratory QA;
  - Oversee laboratory QA/QC documentation;
  - Oversee detailed laboratory data review;
  - Decide laboratory corrective actions, if required;
  - Present technical laboratory QA procedures; and
  - Ensure that the laboratory protocols specified in this QAPP are followed.

3. Laboratory Responsibilities

- CompuChem and Air Toxics Laboratory Project Managers – The Laboratory Project Managers will:

  - Coordinate laboratory analyses,
  - Supervise in-house chain-of-custody,
  - Oversee laboratory data review,
  - Oversee preparation of analytical reports, and
  - Approve final analytical reports prior to submittal to ENVIRON.

- Independent Laboratory Data Validation – Data validation will be provided by MAKuehl Company of Green Bay, Wisconsin or approved alternate data validation firm.

The primary responsibility for project quality rests with the Project Coordinator. Independent QA will be provided by the laboratory Project Managers and the QA Director prior to the release of data packages.

4. Field Responsibilities

- ENVIRON Field Activities Coordinator – ENVIRON Field Activities Coordinator, Cynthia Bonczkiewicz, P.E., will be supported by the technical and field staff from ENVIRON and other firms, as needed. The Field Activities Coordinator will be responsible for leading and coordinating the day-to-day activities of the various resource specialists under ENVIRON's supervision and will report directly to the ENVIRON Project Coordinator. Specific Field Activities Coordinator responsibilities include:

  - Coordination of Contractor or specialty contractors in the implementation of field-related plans, and adherence to management-developed requirements;

  - Coordination and management of ENVIRON field staff, including sampling and subcontractors;

  - Implementation of QC for technical data provided by the ENVIRON field staff, including field measurement data;

  - Review of data from ENVIRON's field team efforts; and

  - Identification of problems at the field team level, discussion of resolutions with the Project Coordinator, and provision of communication between project team members and upper management.

C. Problem Definition/Background

1. Site/Facility History

The ECC Site is located in Boone County, north of Zionsville, Indiana, approximately 10 miles northwest of Indianapolis, in an area that is primarily agricultural

but that also contains some areas of commercial and industrial land use. There are no current operations at the ECC Site. Storage and/or disposal operations have not been conducted since approximately 1983.

Between 1987 and 1990, field investigations of the Site were conducted by Environmental Resources Management-North Central, Inc. (ERM-North Central), on behalf of the ECC Potentially Responsible Parties, and CH2M Hill. The results of the soil and ground water investigations indicated that the primary significant chemical constituents found at the ECC Site are chlorinated volatile organic compounds (VOCs). Remediation activities, including excavation of the Southern Concrete Pad area and installation of a SVE system on the North and Central Treatment Areas, were conducted from 1997 through 2000.

As presently configured, the SVE system that has been installed at the ECC Site has not achieved the subsurface water cleanup standards in the till, which are set forth in Table 3-1 to Revised Exhibit A (included in the attached Design Report as Table 2-1). USEPA and IDEM are concerned that failure to achieve those cleanup standards may, over time, have an adverse effect on water quality in Unnamed Ditch, which is located adjacent to the eastern portion of the Site. For that reason, Revised Exhibit A, the Consent Decree, and the amended Record of Decision[1] (ROD) provide for specific Additional Work to be performed if USEPA determines that those standards were not met within a 5-year period, unless the parties agree otherwise.

These standards were not met within the 5-year period provided in the Consent Decree. The agreed modifications to the "Additional Work" provisions of Revised Exhibit A and the Consent Decree were presented in Attachment Z-1.

2. Project Description

The Attachment Z-1 Remedy includes an augmented soil vapor extraction (SVE) system that augments the existing SVE system by installing additional SVE trenches generally along the alignment of a ground water collection trench previously required as Additional Work in Revised Exhibit A to the Consent Decree. The new SVE trenches will be connected to the existing SVE system and will be operated using all of the basic operations of the existing SVE system equipment. In order to provide additional protection to Unnamed Ditch, the Attachment Z-1 Remedy also includes a perimeter thin barrier curtain wall (TBCW), which was constructed in May 2006, and a permeable reactive gate system (PRGS). The Attachment Z-1 Remedy enhances and replaces the water interception

[1] The original ROD for the Site was issued in September 1987, and the Amended ROD was issued in June 1991.

trench originally required as the Additional Work in Revised Exhibit A. The Attachment Z-1 work will be conducted under the Additional Work provisions of the Consent Decree, as amended.

After construction of the augmented SVE trenches and the PRGS is completed, there will be several distinct phases for the operation of the modified Additional Work. The activities will be different for each period. The periods and the associated activities are as follows:

- **Active Phase**. This is defined as the period of operation of the augmented SVE trench system.

- **Phase I Monitoring**. This is defined as the 1-year period beginning when the Soil Vapor Standards have been achieved in the augmented SVE trenches. At the completion of the Phase I Monitoring period, Phase II Long-Term Monitoring will begin at the Site.

- **Phase II Long-Term Monitoring**. This is defined as the period following the completion of Phase I Monitoring. It is divided into Phase II(a) and Phase II(b).

D. Project/Task Description and Schedule

1 Project Objectives

Augmented SVE system activities will be conducted to alleviate the potential and actual threats to human health and the environment posed by the hazardous substances at the ECC Site. As presently configured, the SVE system that has been installed at the ECC Site has not achieved the subsurface water cleanup standards in the till set forth in Table 3-1 to Revised Exhibit A (included in the attached Design Report as Table 2-1). The Consent Decree and the amended ROD provide for specific Additional Work to be performed. The objective of this Attachment Z-1 Remedy is to meet the standards referenced above.

2. Project Target Parameters and Intended Data Usages

Sample matrices and analytical parameters are presented in Table E-1. The field parameters are basic water quality parameters to be obtained during water and soil gas sampling conducted during and following augmented SVE system operations.

Field Parameters

The field parameters to be measured include screening of stockpiled soils excavated from the augmented SVE trenches or organic vapors using a photoionization detector (PID), analysis of extracted soil vapors for total organic compounds using a continuous vapor analyzer, certain field tests to confirm biopolymer breakdown, and field parameters measured from various water samples.

Laboratory Parameters

The analytical parameters and methods to be followed for all sampling activities are presented in Table E-1. Method detection limits (MDLs) and/or reporting limits (RLs)/instrument detection limits (IDLs) are provided in Tables E-2, E-3, E-4, and E-5.

3. Project Schedule

A preliminary work schedule will be submitted as a Contractor Submittal. The actual time to implement the Attachment Z-1 Remedy may vary, depending on changes in scope, USEPA review periods, or other factors. The project schedule will be revised accordingly throughout the project. The actual date of project mobilization is dependent on USEPA approval of the Design Report and submittal of Contractor Submittals.

E. Quality Objectives and Criteria for Measurement Data

The overall QA objective is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide high quality results, and which are legally defensible in a court of law. Specific procedures for sampling, chain of custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. Standard Operating Procedures (SOPs) for laboratory analyses are provided in Attachment E-1 (soil and ground water) and Attachment E-2 (soil vapor and air). The SOPs address the required accuracy, precision, and sensitivity of the analyses. The SOPs also provide the laboratory analytical sensitivity requirements for the analytical methods and parameters.

1. Data Quality Objectives (DQOs) Process

DQOs are qualitative and quantitative statements that clearly state the objective of a proposed project, define the most appropriate type of data to collect, determine the most appropriate conditions for data collections, and specify acceptable decision error limits that establish the quantity and quality of data needed for decision making. The DQO process is described in a series of seven sequential steps: Step 1: Stating the Problem; Step 2: Identifying the Decisions; Step 3: Identifying Inputs to the Decisions; Step 4: Defining the Boundaries of the Study; Step 5: Developing a Decision Rule; Step 6: Specifying Limits on Decision Errors; and Step 7: Optimizing the Design. The DQO process is discussed below, including applicable aspects of the data collection activities associated with the Attachment Z-1 Remedy. Certain information contained in other portions of the Design Report is included by reference.

Step 1: Stating the Problem

In general, the augmented SVE system is being installed to reduce contamination levels in subsurface till water by removing contaminants using SVE until the soil vapors meet the site-specific Soil Vapor Standards provided in Table 2-2 of the Design Report. A thin barrier curtain wall (TBCW) and associated piezometers are already installed. The remaining work includes: (1) construction of seven augmented SVE trenches; (2) dewatering of the trenches; (3) operation of the augmented SVE system; (4) soil vapor sampling, as necessary, to confirm completion of augmented SVE activities; (5) sampling of subsurface water; (6) augmented SVE trench water; and (7) surface water to confirm compliance with the Acceptable Stream Concentrations. In addition, excavated soils will be sampled to determine whether on-site treatment, as set forth on Figure 2-1 of the Design Report, is necessary. Other data gathering will involve a dewatering test during the Construction Phase.

The roles and responsibilities of all key management, QA, laboratory and field personnel are provided in Section I.B above. The DQOs for the project have been developed by ENVIRON personnel (the "scoping team") under the direction of the ENVIRON Project Coordinator for the ECC Trust. The DQOs are based on the USEPA-approved ECC Site Acceptable Concentrations contained in Table Z-1-1 of Attachment Z-1, which is included as Table 2-1 of the Design Report.

Step 2: Identifying the Decisions

Field observations will confirm that the design plans are effectively carried out. Field sampling will confirm the effectiveness of the design and/or remedial activities. The criteria for moving from the Active Phase to the Phase I Monitoring Phase and then the Phase II Long-Term Monitoring Phase are described in the Design Report. The results of the soil vapor sampling activities will determine the start of Phase I Monitoring Phase. The results of surface and monitoring well water analyses will determine the start of the Phase II Long-Term Monitoring Phase.

The following is a summary of the decisions to be made for each environmental sampling activity:

- **Confirmation of End of Active Phase**. The augmented SVE system will be operated until the vapor concentrations in all augmented SVE trenches are less than the Soil Vapor Standards. When the results of analyses of the combined soil vapor sample collected from two consecutive restart spikes conducted two weeks apart show that the concentrations meet the Site-Specific Acceptable Concentrations, a water sample will be collected from the augmented SVE system. If the water sample meets the Acceptable Stream Concentrations, the operation of the augmented SVE system will be terminated.

- **Completion of Phase I Monitoring**. At the end of the 1-year Phase I Monitoring period, the water level and ground water concentration data from the designated piezometers/monitoring wells will be evaluated. If the ground water and surface water meet the Acceptable Stream Concentrations and the TBCW is performing as designed, Phase I Monitoring will be considered complete.

- **Phase II Monitoring**. The first two years (Phase II (a)) will include quarterly sampling/analysis of water from the individual trench segments and selected sand and gravel wells. If the subsurface and surface water meets the Acceptable Stream Concentrations after 2 years, Phase II(a) is complete. Phase II(b) Long-Term Monitoring will include annual maintenance of the permeable reactive gate system (PRGS) system and monitoring of the PRGS effluent. This will continue until the USEPA, in consultation with IDEM, agrees to suspend the requirement.

Step 3: Identifying Inputs to the Decisions

The types of information that will be used as input to the decision consists of: (1) the Design Report; (2) Attachment Z-1; (3) data generated from field analysis of soil, soil vapor, and ground water; (4) analytical data generated from laboratory analysis of soil vapor, ground water and surface water; and (5) on-site instrumentation and field observations of the dewatering/SVE activities (pressure gauges, flow meters, etc.).

Field data that will be used as input to the decisions include the following:

- TBCW piezometer water levels,
- Pumping rate and water level data from initial trench segment dewatering tests, and
- Ground water and surface water analysis results.

Step 4: Defining the Boundaries of the Study

The general spatial boundaries of the Attachment Z-1 Remedy include the ECC property boundaries and Unnamed Ditch, which is adjacent to the ECC Site. Attachment Z-1 Remedy activities will be limited to the east, south, and southwest sides of the Site. Ground water samples will be collected from the designated monitoring wells (on site) and the PRGS (on site). Surface water samples will be taken from Unnamed Ditch just east and south of the Site (i.e., at off-site locations).

At this time, no temporal boundaries that could affect the decision are anticipated. With the possible exception of meteorological conditions, which could affect the timing or methods of sample sample collection activities, and site access needs, no practical constraints on data collection are envisioned.

Step 5: Developing a Decision Rule

The purpose of developing a decision rule is to integrate the output from previous steps of the DQO process into a statement that defines the parameter of interest; delineates the scale of decision making; specifies the action level; and describes the logical basis for choosing among alternative actions. The output from this step is an "if...then..." statement that defines the conditions that would cause the decision maker to choose among alternative courses of action. All decision rules are based on meeting the action levels approved in Attachment Z-1. Decision rules for each stage of the Attachment Z-1 Remedy that is

impacted by environmental sampling and analysis, including alternative actions, are discussed in the Design Report.

Step 6: Specifying the Limits on Decision Errors

The Design Report and the sampling and analysis program discussed in the Addendum to the Field Sampling Plan provide procedures for obtaining Site data. Corrective actions may be considered during implementation of the Attachment Z-1 Remedy, if suspect or questionable laboratory or field data are identified through QA/QC reviews.

As stated in the IDEM RISC Technical Guide, a Type I Decision Error occurs when the Null Hypothesis is rejected when it is true (False Positive), and Type II Decision Error occurs when the Null Hypothesis is not rejected when it is false (False Negative). In the IDEM RISC Technical Guide, the Null Hypothesis is defined as the theory to be tested. For environmental evaluations, the Null Hypothesis is generally that the site is contaminated. However, for the subject Attachment Z-1 Remedy, the Null Hypothesis is that the relevant Acceptable Concentrations have been achieved through implementation of the ASVES. A decision/goal diagram is attached as Figure E-2.

As discussed in the Design Report, each phase of the Attachment Z-1 Remedy requires the achievement of an Acceptable Concentration for the respective corrective action phase. Therefore, while Type I errors may exist at some point during implementation of the work, only Type II errors (False Negatives) are relevant to the decision making process (e.g., the hypothesis that action levels have been achieved based on sampling data is in fact false). Sampling design error and measurement error will be minimized through such practices as: multiple confirmation sampling events (two augmented SVE system restart tests), field and laboratory QA/QC practices, field audits, and data validation. Statistical tests recommended in the RISC Technical Guide limit Type II decision errors to at least a 25% probability. This decision error limit is appropriate for all Attachment Z-1 Remedy sampling and analysis activities.

Step 7: Optimizing the Design

The design of the field activities will be evaluated during the finalization of the Design Report, as well as during the implementation, operations, and monitoring phases of the Attachment Z-1 Remedy. Proposed revisions will be submitted, in writing, to the USEPA for approval.

2. Precision

Definition

Precision refers to how closely two or more measurements of the same parameter or property agree with each other. Chemical concentration data obtained from the analyses of field duplicate and matrix spike duplicate samples will be compared to evaluate analytic precision. Field precision will also be determined by the collection of duplicate PID and any other field screening measurements.

Field Precision Objectives

Duplicate air, surface water, and ground water samples will be collected in the field. The project goal is to collect 1 duplicate per 10 samples during the monitoring/confirmation sampling. Section II.E of this Plan describes duplicate collection techniques and review. QA objectives for field parameters are presented in Table E-6.

Laboratory Precision Objectives

Measurement of precision is mathematically defined for laboratory analysis in Section IV.A of this QAPP. Project QA objectives for laboratory parameters are as presented in the laboratory SOPs contained in Attachments E-1 and E-2.

3. Accuracy

Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value.

Field Accuracy Objectives

The accuracy of the field data will be maintained by ensuring that instruments are in good working condition and properly calibrated. Each piece of equipment will have a unique serial number for tracking during field use, calibration, and maintenance. Team members will be familiar with the field calibration, operations, and maintenance of the equipment. They will maintain proficiency in equipment operation, perform the prescribed field operating and calibration procedures outlined in the equipment manuals for each respective instrument, and keep records of all

field instrument calibrations and field checks in logbooks. The accuracy of the PID field screening and ground water field parameter measurements will be evaluated in conjunction with the instrument calibration records to ensure the highest possible accuracy. Accuracy in the field will also be assessed through the use of equipment rinse and trip blanks as discussed in Section IV.A of this QAPP and through adherence to all sample handling, preservation, and holding time requirements. QA objectives for field parameters are presented in Table E-6.

Laboratory Accuracy Objectives

Laboratory accuracy is mathematically defined in Section IV.A of this QAPP. These are updated annually based on the laboratory performance data from the previous year. Project Accuracy control limits are as provided in the laboratory SOPs in Attachments E-1 and E-2.

4. Completeness

Definition

Completeness is a measure of the percentage of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained. The equation for completeness is provided in Section IV.A of this QAPP. Field completeness for this project will be greater than 90%.

Laboratory Completeness Objectives

Laboratory completeness is a measure of the percentage of valid measurements obtained. The equation for completeness is presented in Section IV.A of this QAPP. Laboratory completeness for this project will be greater than 90%.

5. Representativeness

Definition

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter, which is dependent on the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of facility conditions.

Measures to Ensure Representativeness of Field Data

Representativeness is a qualitative parameter, which is dependent on the proper design of the various monitoring programs and proper laboratory protocol. The monitoring plans are designed to provide data representative of site conditions. Representativeness will be satisfied by requiring that the procedures detailed in the Addendum to the FSP are followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be ensured by the selection of proper well locations/depths and samples. The well and sample locations are documented in the Addendum to the FSP.

Measures to Ensure Representativeness of Laboratory Data

Representativeness will be satisfied by ensuring that proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory.

6. Comparability

Definition

Comparability expresses the confidence with which one data set can be compared with another. To the extent that the data collection objectives and analytical methods are similar, data generated during previous field investigations are expected to be comparable to data that will be generated.

Measures to Ensure Comparability of Field Data

Comparability is dependent on proper design of the sampling program and will be satisfied by ensuring that the Addendum to the FSP and this QAPP are followed. The methods used to collect field data will be consistent during the Attachment Z-1 Remedy, thereby, ensuring comparability of field data.

Measures to Ensure Comparability of Laboratory Data

The extent to which existing and planned analytical data will be comparable depends on the similarity of the sampling and analytical methods as documented in this QAPP. Comparability is also dependent on similar QA objectives. The laboratories, sample collection methods, and the methods used to analyze samples will be consistent during all phases of the Attachment Z-1 Remedy, ensuring comparability of laboratory data.

7. Level of Quality Control Effort

Trip blank, field blank, field duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the various monitoring programs implemented for this project. Trip blanks will be submitted to the laboratories to provide the means to assess the quality of the data resulting from the program. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Field blanks are used to assess the effectiveness of equipment decontamination procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spike/matrix spike duplicates (MS/MSDs) provide information about the effect of the sample matrix on the measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

One field duplicate will be collected for every 10 or fewer aqueous samples. One MS/MSD will be collected for every 20 or fewer aqueous samples. Aqueous MS/MSD samples must be collected at triple the volume for analysis. One trip blank will be analyzed per shipment of aqueous samples. One field equipment blank will be collected for every 10 or fewer aqueous and soil vapor samples.

The number of field duplicate samples, trip blank samples, field blank samples, and MS/MSDs to be collected is detailed in Table E-1.

F. Special Training/Certification

No specialized training or certification beyond that typically required for this type of work (e.g., engineer or geologist for drilling, Occupational Safety and Health Act (OSHA) health and safety training, etc.) will be needed for the completion of this project.

G. Documents and Records

The Project Coordinator will be responsible for ensuring the appropriate project personnel have the most current approved version of the QAPP. Any revisions to the QAPP will be circulated to the individuals listed in Section I.A.

Detailed information concerning the field and laboratory reporting and recordkeeping procedures is provided in Section II.J. The content of and reporting frequency for project QA reports is discussed in Section III.B.

The final project file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this QAPP. The Project Coordinator is custodian of the final project file and maintains the contents of evidence files, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews in a secured, limited access area and under custody of the Project Coordinator. The final disposition schedule and location will be determined by the Project Coordinator during the Phase II(a) Long-Term Monitoring period.

II. GROUP B: DATA GENERATION AND ACQUISITION

A. Sampling Process Design

The field sampling program is discussed in the Design Report and the Addendum to the FSP. Table E-1 presents a summary of this information.

B. Sampling Methods

1. Sample Collection/Preparation Procedures

The sample collection procedures are detailed in Section III of the Addendum to the FSP.

2. Sample Containers, Preservatives, and Volume Requirements

Requirements for sample containers, preservation and volume are summarized in Table E-7.

3. Decontamination Procedures

Decontamination procedures are described in Section III.J of the Addendum to the FSP.

4. Sample Packaging and Shipment Procedures

As soon as sample labeling is completed, each soil or water sample will be placed in an insulated container (cooler) that contains sufficient ice to maintain the sample temperature between 4 degrees Celsius (^{o}C) and 6 ^{o}C. During each sampling task, samples will be shipped to the fixed-base laboratory at least every other working day. The sample containers will be placed in ZipLoc^{TM} bags and then individually wrapped with cushioning material (e.g., bubble wrap). The bottom of the insulated sample shipping container (cooler) will be lined with cushioning material (e.g. bubble wrap) to reduce the possibility of sample container breakage. Sufficient ice will be placed in each cooler to maintain the temperature of the samples between 4 ^{o}C and 6 ^{o}C for the overnight shipment to the fixed-base laboratory. Chain-of-custody procedures will be followed as described in Section II.C. Once the required chain-of-custody activities (forms, custody seals, etc.) have been performed, the shipping container will be additionally sealed with plastic shipping tape, and shipped via overnight courier for morning delivery to CompuChem Laboratories.

Soil vapor samples will be handled as listed above except that the Summa canisters will not be individually wrapped and no ice is required. Soil vapor samples will be shipped to Air Toxics Laboratories.

5. Field Corrective Action

Corrective action in the field may be needed when the sample network is changed (e.g., more/fewer samples, sampling locations other than those specified in the QAPP, etc.) or when sampling procedures require modification due to unexpected conditions. Field and technical staff will be responsible for reporting suspected technical or QA non-conformances or suspected deficiencies of any field activity or issued document by reporting the situation to the ENVIRON Field Activities Coordinator, or designee. The ENVIRON Field Activities Coordinator, or designee, will be responsible for assessing the suspected problems with ENVIRON activities in consultation with the Project Coordinator and/or QA Director. A decision will be made based on the potential for the situation to impact the quality of the data. The ENVIRON Field Activities Coordinator, or designee, will then be responsible for initiating corrective action for non-conformances by:

- Evaluating all reported non-conformances,
- Determining disposition or action to be taken,
- Maintaining a log of non-conformances,
- Reviewing non-conformance reports and corrective actions taken,
- Maintaining a log of non-conformances and follow-up testing after corrective actions are complete, and
- Verifying that non-conformance documentation is included in the project files.

If appropriate, the Project Coordinator, or designee, will see that no additional work that is dependent on the non-conforming activity is performed until the corrective actions are completed.

The ENVIRON Field Activities Coordinator, or designee, is responsible for controlling, tracking, and implementing the identified changes. All significant changes will be reported by the Project Coordinator in the monthly progress reports submitted to USEPA.

C. Sample Handling and Custody

Custody is one of several factors that is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements

for admissibility: relevance and authenticity. Sample custody is addressed in three parts: (1) field sample collection, (2) laboratory analysis, and (3) final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if it is:

- In your possession;
- In your view, after being in your possession;
- In your possession and you place it in a secured location; or
- In your designated secure area.

A chain-of-custody record is a record of all persons who have collected, relinquished, and/or received samples and the dates and times when these activities occurred. Items that must be held under a chain of custody include samples, sample tags, airbills and a chain of custody record form. The chain of custody will be initiated in the field and will be maintained until delivered to the laboratory. The laboratory is a restricted access facility with lockable cold storage. Sample containers that are returned to the field or subcontracted shall initiate the chain-of-custody procedure again.

1. Field Custody Procedures

The sample packaging and shipment procedures, summarized below, will ensure that the samples will arrive at the laboratory with the chain of custody intact. The protocol for specific sample labeling is included in Section III and Attachment D-8 of the addendum to the FSP. Examples of the Air Toxics and CompuChem chain of custody forms, sample labels, and custody seals are included in Attachment E-3.

Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples. The following are basic sampling procedures:

- All containers will be labeled with sample numbers and locations, date/time of collection, and type of analysis. The sample numbering system is presented in Section III.I of the Addendum to the FSP.

- A sample label will be attached to each individual sample aliquot for each investigative QC sample. The sample label will include the following information: the field sample number, date and time of collection, type of analysis, type of preservative (if any), a space for the laboratory sample number, project identification, and the name of the person collecting the sample. The label may also include a space for comments. The sample label will be adhesive-backed and will be attached to the sample container. Sample labels will be completed for each sample using waterproof ink.

- The ENVIRON Field Activities Coordinator and Project Coordinator will review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

Transfer of Custody and Shipment Procedures

Samples collected for off-site analysis at the CompuChem or Air Toxics fixed-base laboratories will be accompanied by properly completed chain-of-custody forms. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the permanent laboratory, or to/from a secure storage area.

Samples will be properly packaged on ice to maintain the sample temperature between 4 °C and 6 °C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record secured to the inside top of each sample box or cooler. Shipping containers will be secured with shipping tape and custody seals for shipment to the laboratory. The cooler will be strapped shut with shipping tape in at least two locations.

The chain-of-custody record, identifying the contents, will accompany all shipments. The original record will accompany the shipment, and the sampler will retain copies.

Airbills or bills of lading will be used when samples are sent by Federal Express, UPS, Airborne Express, or Express Mail. Receipts of airbills or bills of lading will

be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Since the custody forms will be sealed inside the sample cooler and coolers will be sealed intact, the commercial carriers will not be required to on the custody form.

2. Laboratory Custody Procedures

All samples shipped off-site for analysis will be received at CompuChem or Air Toxics by the sample custodian. It will be the responsibility of the sample custodian to determine whether or not the samples are close to exceeding their holding time and require immediate attention and the manner in which those samples will be split, preserved, stored, or routed.

The sample custodian is responsible for the receipt, log-in, and access controlled storage of all client samples. Each sample is labeled with a unique number, which is entered into the sample receiving log. The samples are placed into appropriate storage within the access-controlled location. All samples are maintained under proper storage conditions for 30 days past the generation of the analytical report.

D. Analytical Methods

All soil, sediment, residue, surface water and ground water samples collected during field sampling activities will be analyzed using USEPA SW-846 methodologies by CompuChem. The soil vapor samples will be analyzed by Air Toxics using Method TO-15 by Air Toxics.

1. Field Analytical and Measurement Procedures

Field methodologies for the collection of field screening data (pH, specific conductance, and temperature) are provided in the Addendum to the FSP. QA objectives for field parameters are included in Table E-6.

2. Laboratory Analytical and Measurement Procedures

SOPs have been prepared for all methods used for analysis of samples for this project. The laboratory SOPs are included in Attachments E-1 and E-2.

Each of the SOPs is based on an analytical method published by the USEPA. Each specifies:

- Procedures for sample preparation,
- Instrument start up and performance check,
- Initial and continuing calibration check requirements,
- Specific methods for each sample matrix type, and
- Required analytical procedures.

The laboratory SOPs in Attachments E-1 and E-2 provide the quantitation limits and QA objectives for the laboratory parameters.

3. Laboratory Corrective Action

Corrective actions may be required for two classes of problems: analytical or equipment problems and noncompliance problems. Specific corrective actions are presented in the laboratory SOPs for each analytical method. Specific corrective actions are not repeated in the text of this QAPP to avoid redundancy.

Analytical or equipment problems may occur during sample preparation, laboratory instrumental analysis, or data review. Corrective measures for these types of problems are discussed in the following sections.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Laboratory QA Officer or Project Coordinator. Implementation of corrective action for noncompliance problems will be confirmed in writing through the same channels.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy,
- Blanks contain target analyses above acceptable levels,
- Undesirable trends are detected in spike recoveries or Relative Percent Difference between duplicates,
- There are unusual changes in detection limits,

- Deficiencies are detected by the laboratory QA Officer during internal or external audits or from the results of performance evaluation samples, or

- Inquiries concerning data quality are received.

In addition, the trip blanks will be monitored for contamination, and corrective actions will be taken as soon as a problem is identified. This will be accomplished either by:

- Discontinuing the use of a specific bottle lot;

- Contacting the bottle supplier(s) for re-testing the representative bottle from a suspect lot,

- Re-sampling the suspected samples,

- Validating the data taking into account that the contaminants could be introduced by the laboratory (e.g., common laboratory solvents, sample handling artifacts, etc.); or

- Evaluating the potential for bottle QC problem, so as to make an educated determination of whether the bottles and hence the data are still usable.

For this particular project, the corrective actions will be conducted in a comprehensive manner in order to avoid the use of identified contaminated lot(s), and to ensure that the bottle supplier(s) is deemed responsive and able to provide clean bottles as specified.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, and checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Project Manager or Operations Manager. Once resolved, full documentation of the corrective action procedure is filed with the Laboratory QA Director.

E. Quality Control

The number of duplicate and blank samples to be collected is listed in Table E-1. Sample procedures are specified below. The evaluation protocols and acceptance criteria for field duplicate samples, split samples, and trip blanks are stated below.

1. Field Duplicate and Split Samples

Field duplicates or split samples should be identified. Reported results for each sample and duplicate will be compared by calculating the relative percent difference (%RPD), as defined in Section IV.A. Positive results for a target compound will be flagged "J" in the sample and its duplicate or split if the following criteria are not met:

- A control limit of +/-20% for aqueous samples (40% for solid samples) for the %RPD will be used if both the sample and its duplicate or split results are greater than 5X the Contract Required Quantitation Limit (CRQL).

- A control limit of +/-40% for aqueous samples (60% for solid samples) for the %RPD will be used if both the sample and its duplicate or split results are less than 5X the CRQL.

2. Field Duplicate Collection

The general level of the QC effort will be 1 field duplicate for every 10 or fewer aqueous samples. The field duplicate samples will be analyzed for the same list of parameters as the ground water, surface water, or soil sample with which they are collected.

Duplicate samples will be collected in accordance with the general procedures described below:

- The investigative sample location from which a duplicate sample will be collected will be identified.

- The duplicate sample will be collected at the same time and location as the investigative sample using the same procedure outlined in the sampling procedure for investigative samples.

- The investigative sample containers for VOC and semivolatile organic compound (SVOC) analyses (or to be analyzed for site-specific constituents of concern) will

be completely filled and sealed, and then the duplicate sample container will be filled and sealed. For all other sample parameter containers, the duplicate and investigative sample containers will be alternately filled.

- The field notebook, sample log sheet, labels, and chain-of-custody forms will be filled out with the duplicate sample properly designated and logged.

- The duplicate sample will be preserved, handled, and shipped following the same procedures as the investigative samples.

3. Matrix Spike/Matrix Spike Duplicate Collection

MS/MSD samples are investigative samples. Aqueous MS/MSD samples must be collected at triple the volume for VOCs. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples. Samples designated for MS/MSD analysis will be collected following the same procedure as other investigative samples, except that additional volume for the aqueous samples will be collected, as necessary. QA objectives for laboratory parameters are included in the SOPs contained in Attachments E-1 and E-2.

4. Field Equipment Blank Collection

To determine the effectiveness of the decontamination procedures, field equipment blank samples will be collected during subsurface water, surface water, and soil vapor sampling. Field equipment blank samples will be collected at a rate of 1 for every 10 or fewer investigative samples for each matrix. To determine the effectiveness of the decontamination procedures for ground water sampling equipment, deionized/distilled water will be poured through a clean bailer or pumped through a peristaltic pump with clean tubing into a laboratory-supplied sample container. For surface water sampling devices, the effectiveness of the decontamination procedures will be evaluated by pouring deionized or distilled water into the device before it is poured into a laboratory-supplied sample container. For soil vapor sampling, the field equipment blank will be taken from ambient air with the sampling apparatus attached.

5. Trip Blank Preparation

Trip blanks are deionized organic-free water samples in VOC vials provided by the analytical laboratory and stored with VOC sample containers before filling and during shipment. These samples will remain unopened. If these "blanks" contain detectable

concentrations of for one or more compounds, the problem could be cross-contamination between sample and container via air in the storage or shipment containers, or laboratory contamination. One VOC trip blank consisting of deionized organic-free water will be prepared at the CompuChem Laboratory and included along with each shipment of aqueous VOC samples.

6. Laboratory QC Procedures

The laboratory SOPs include a QC section that addresses the minimum QC requirements for the analysis of specific analyte groups. For this project, no specific compounds will be added to the spiking solution; rather standard QA/QC measures will be implemented.

If it cannot be demonstrated that the selected compounds are being analyzed in an accurate and precise manner, the analytical approach will be reviewed and changed, as necessary. An example of a potential change is modification of the matrix spike (MS) solution to include the poor purging compounds. All field QC samples to be collected are described above and in the Addendum to the FSP.

F. Instrument/Equipment Testing, Inspection, and Maintenance

1. Field Instrument Preventive Maintenance

A PID and water quality meters will be used during the subsurface sampling. Specific preventative maintenance procedures to be followed are those recommended by the manufacturer. No internal calibration or maintenance procedures will be conducted in the field; these procedures are solely performed by the instrument supplier. These instruments will be checked daily and calibrated at an interval recommended by the manufacturer. Backup instruments and equipment will be available on site or within 1-day shipment to avoid delays in the field schedule.

2. Laboratory Instrument Preventive Maintenance

As part of their QA/QC program, a routine preventive maintenance program is conducted by CompuChem and Air Toxics to minimize the occurrence of instrument failure and other system malfunctions. Laboratory personnel perform preventive maintenance and repair or coordinate with a vendor for the repair of their instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance is carried out on a regular,

scheduled basis, and is documented in the laboratory instrument service logbook, for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided under a repair and maintenance contract with factory representatives.

G. Instrument/Equipment Calibration and Frequency

This section describes procedures for maintaining the accuracy of all instruments and measuring equipment used for conducting field and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled, periodic basis.

1. Field Instrument Calibration

The PID and water quality meters shall be calibrated at the interval specified in the manufacturer's instructions, and all calibration data shall be recorded in dedicated calibration logbooks or field logbooks. Only calibration gases provided by the equipment supplier will be used. Deionized water will be obtained from commercial suppliers. Potable water will be obtained from the city water supply.

The sample containers used for this project will be prepared in accordance with the USEPA, 1993 Guidance Document, *Specification and Guidance for Contaminant-Free Sample Containers* (EPA 540/R-93/051). Tedlar™ bags, canisters, vials, jars, and bottles used for sampling will not contain contaminants exceeding the level specified in the above-mentioned document. The laboratory container supplier will provide a certificate of cleanliness on request.

2. Laboratory Instrument Calibration

Calibration of laboratory equipment will be based on approved written procedures contained in the Laboratory SOPs (Attachments E-1 and E-2) or the appropriate analytical methods. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory analyst. These records will be filed at the location where the work is performed and will be subject to QA audit. The laboratory maintains in-house spare parts and/or service contracts with vendors.

H. Inspection/Acceptance of Supplies and Consumables

Consumable field items will be inspected by the ENVIRON Field Activities Coordinator, or designee, prior to acceptance and use.

I. Non-Direct Measurements

No data needed to complete this project will be obtained from non-measurement sources.

J. Data Management Procedures

1. Field Data Reporting

Field logbooks will provide the means of recording data collection activities. As such, entries will describe procedures in as much detail as possible so that persons going to the ECC Site could reconstruct a particular situation without reliance on memory. Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned,
- Logbook number,
- Project name,
- Project start date, and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

A record will be kept of field measurements and collected samples. All entries will be made in ink, signed and dated, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark signed and dated by the sampler. The number of photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified.

The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, sample volume, and number of containers. The sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample

identification number, will be noted under sample description. The system for assigning a sample identification number is described in the Addendum to the FSP.

Field data reporting shall be conducted principally through the transmission of spreadsheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities. If field logbook information is to be used in the project reports, it will likely be presented in a tabular format.

2. Laboratory Data Reporting

Upon acceptance of the preliminary reports by the Laboratory QA Officer, final reports will be generated and signed by the Laboratory Project Manager. The laboratory package shall be presented in the same order in which the samples were received.

CompuChem/AirToxics will prepare and retain full analytical and QC documentation similar to that required by the contract laboratory program. Such retained documentation need not be hard (paper) copy, but may be in other storage media (e.g., electronic). The laboratories will supply a hard copy of the retained information on an as needed or as requested basis.

CompuChem will report the data in the same chronological order in which analyses are conducted, along with QC data. Each analytical data package will include the following:

- Cover sheets (Signature Pages) listing the samples included in the report and narrative comments describing problems encountered in analysis;
- Tabulated results of organic compounds identified and quantified for investigative and blank samples (such as Form I from LIMs);
- Analytical results for sample spikes, sample duplicates, and laboratory control samples; and
- Tabulation of instrument detection limits determined in pure water.

For organic analyses, surrogate spike recoveries, chromatograms, gas chromatography/mass chromatography (GC/MS) spectra, calibration verification of standards and blanks, GC/MS system tuning data, standard procedural blanks, and raw data system printouts (or legible photocopies) identifying date of analyses, analyst's name, and parameters determined, will be retained by the laboratory. The complete laboratory data

package will be provided to MAKuehl Company or approved alternate for the performance of data validation.

All analytical data generated for this removal action will be computerized in a format organized to facilitate data review and evaluation. The ECC data set will be available for controlled access by the Project Coordinator and by authorized personnel. The final data deliverables will be presented in a "CLP-like" format.

III. GROUP C: ASSESSMENT AND OVERSIGHT

A. Assessments and Response Actions

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of QC performance, which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective action proposed and implemented should be documented in the regular QA assurance reports to management. Corrective action should only be implemented after approval by the Project Coordinator, or designee.

For non-compliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Laboratory QA Officer or Project Coordinator, as appropriate. Any non-conformance with the established QC procedures in this QAPP or Addendum to the FSP will be identified and corrected in accordance with this QAPP.

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the Addendum to the FSP and the QAPP. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

1. Field Performance and System Audits

Internal Field Audits

Field Sampling Technical System Audits (TSAs) of field activities will be conducted by the QA Director or a designated representative. One TSA will be conducted during the initial phase of the field sampling activities to verify that all established procedures are followed. In addition, another field TSA will be performed if the sampling team or procedures are changed during the monitoring period. The TSA will include a Field Analytical Audit, including an audit of PID and water quality measurement procedures. The need for additional internal audits will be determined by the QA Director. An example of a situation that would likely trigger an audit would be difficulties in obtaining reproducible laboratory data that could not be attributed to laboratory procedures. In such a situation, an internal audit of field sampling procedures may be warranted to assist in identifying the cause of the difficulties. Another example of a situation where an internal audit may be

conducted would be following the implementation of significant corrective action activities.

The field sampling TSAs will be conducted by the QA Director, Field Activities Coordinator, or designee. The audits will include examination of field sampling and measurement records; field instrument calibration and operating records; and sample collection, handling, packaging, and documentation in compliance with the established procedures.

External Field Audits

An external audit may be conducted at the discretion of the USEPA/IDEM Project Manager. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the USEPA/IDEM. External field audits will be conducted according to the field activity information presented in the QAPP.

2. Laboratory Performance and Systems Audits

Internal Laboratory Audits

The internal performance and system audits of CompuChem/Air Toxics will be conducted by the CompuChem and Air Toxics QA Directors. The system audits, which will be done on an annual basis, will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedure, sample preparation and analysis, and instrument operating records.

The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, and instrument operating records. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The QA Directors will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

External Laboratory Audits

Any external audits may be conducted at the discretion of the USEPA/IDEM Project Manager.

B. Reports to Management

TSA audit results will be provided in writing to the Project Coordinator within 2 weeks of completion of the audit. In addition, the ENVIRON QA Director and field personnel will report any field QA difficulties or lack of compliance to the Project Coordinator as specified in Section III.A. Issues will include any problems regarding sampling, field measurement, sample handling, communication, or documentation. The laboratory QA Director will report any laboratory QA problems to the Project Coordinator. The Project Coordinator will maintain a file of any checklists or corrective action letters during the project.

Report(s) containing field sampling data will contain separate QA sections in which data quality information collected during the task is summarized. The data report(s) will be the responsibility of the Project Coordinator and will include the appropriate laboratory QA Officer reports on the accuracy, precision, and completeness of the data, as well as the results of the TSAs, and any corrective action needed or taken, provided analytical data were generated for the report.

The QA sections of the data report(s) will contain a discussion of the QA issues noted above. It will also include a discussion of any qualified data, and major project problems. The section will also provide justification for use of qualified and/or if necessary, recommend that supplemental data be collected to replace the affected data set.

IV. GROUP D: DATA VALIDATION AND USABILITY

A. Data Review, Verification, and Validation

All chemical analysis data generated will be reviewed based on its accuracy, precision, and completeness. The following data will be validated as defined in the following sections:

- Soil vapor from the augmented SVE system following two restarts spike tests;
- Combined ground water sample from PRGS manifold manhole following two successful restart spike tests;
- Phase I Monitoring sampling, at the end of the one-year monitoring period; and
- Phase II(a) Long-Term Monitoring, at the end of the first 2 years of the monitoring period.

For those data sets listed above, Data Validation Reports in a "CLP-like" format will be prepared along with the data.

1. Accuracy Assessment

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section II.E using the analytical results of method blanks, reagent/preparation blanks, MS/MSD samples, equipment rinse blanks, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using:

$$\%R = [(A - B) / C] \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample
B = the background level determined by a separate analysis of the unspiked sample
C = the amount of the spike added

2. Precision Assessment

Precision of laboratory analyses will be assessed by comparing the analytical results between duplicate samples (investigative and MS/MSD). The (%RPD) will be calculated for each pair of duplicate analyses using:

$$\%RPD = (S - D) / [(S + D) / 2] \times 100$$

Where:

S = First sample value (original or MS value)

D = Second sample value (duplicate or matrix spike duplicate [MSD] value)

3. Completeness Assessment

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision-making. The completeness is calculated using:

$$\text{Completeness} = [(\text{valid data obtained}) / (\text{total data planned})] \times 100$$

B. Verification and Validation Methods

1. Procedures Used to Validate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field logbooks, on the part of field crew members. This task will be the responsibility of the Field Activities Coordinator.

2. Procedures Used to Validate Laboratory Data

CompuChem/Air Toxics will perform in-house analytical data validation under the direction of the Laboratory QA Director. The Laboratory QA Directors are responsible for assessing data quality and advising of any data that were rated "preliminary or unacceptable" or other notations that would caution the data user of possible unreliability.

Data validation by the laboratory will be conducted as follows:

- The Laboratory QA Director will complete a thorough audit of preliminary reports at a frequency of 1 in 10.

- The Laboratory QA Director and area supervisors will decide whether any sample re-analysis is required.

Assessment of laboratory data will be accomplished by the joint efforts of their respective Laboratory QA Officers and Laboratory Project Managers. The data assessment

by the Laboratory Project Manager will be based on the assumption that the sample was properly collected and handled according to the FSP and Section IV of this QAPP.

CompuChem/Air Toxics data reviewers will conduct a systematic review of the data for compliance with the established QC criteria based on the spike, duplicate, and blank results provided by the laboratory. An evaluation of data accuracy, precision, sensitivity, and completeness based on criteria discussed in Section IV.A of this QAPP will be performed.

Independent data validation will be performed for the sampling events listed above in Section IV.A. MAKuehl Company, or approved alternate, will evaluate the data using guidance from the following EPA documents:

- *National Functional Guidelines for Organic Data Review* (dated October 1999);
- *Standard Operating Procedures (SOPs) for Validation of CLP Organic Data* (EPA Region V, dated April 1991; revised August 1993, February 1997, and February 1994); and
- *Standard Operating Procedure (SOP) for Validation of CLP Inorganic Data*, (dated September 1993).

The data reviewers will identify any out-of-control data points and data omissions and will interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the Project Coordinator based on the extent of the deficiencies and their importance in the overall context of the project. The Project Coordinator may seek USEPA/IDEM input and approval prior to repeating any sample collection and analysis, as appropriate.

C. Reconciliation with User Requirements

The Project QA Director will review the data validation results to determine the data's accuracy, precision, and completeness objectives as defined in Section IV.A of this QAPP.

The response to address data that does not meet the QC objectives noted above will be based on whether the data is critical or non-critical. Critical data is defined as that which is required to achieve project objectives as follows:

- Determine when the Soil Vapor Standards are achieved,
- Determine when Site-Specific Acceptable Concentrations are achieved for subsurface water,
- Confirm the successful completion of Phase I Monitoring, and
- Confirm successful completion of the 2-year Phase II(a) Long-Term Monitoring Phase.

Should critical data be found to not meet QC objectives, the Project Coordinator will take an appropriate course of action to obtain acceptable data. This may include collecting new investigative samples, re-analyzing existing samples, or other action that will result in obtaining acceptable data. The specific course of action will be determined on a case-by-case basis.

Non-critical data is defined as that which, while providing useful information, is not critical to n completing the project objectives. Non-critical data that does not meet the QC objectives will be appropriately documented; however, re-sampling or re-analysis to address such data will not typically be performed.

V. GROUP E: REFERENCES

USEPA. 1994. USEPA Contract Laboratory Program; National Functional Guidelines for Inorganic Data Review.

USEPA. 1999. EPA Requirements for Quality Assurance Project Plans, Interim Final; EPA QA/R-5. Washington, D.C.

USEPA. 1999. USEPA Contract Laboratory Program, National Functional Guidelines for Organic Data Review.

USEPA. 2000. Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Region 5. Revision 0, based on EPA QA/R-5.

TABLES

TABLE E-1

Sampling Schedule and Analytical Parameters
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix Sampled	Sampling Area	Frequency	Field Parameters	Laboratory Analyses	Laboratory Methods	Duplicates	Trip Blanks	Field Equipment Blanks	MS/MSD
Soil Vapor- Individual Trench Segments	SVE System- Treatment Building at field piping connection to building piping	Daily- 1st week, weekly- next 4 weeks, biweekly thereafter during Active Phase	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor- All Trench Segments Combined	SVE System- Treatment Building at the in-line analyzer	Daily for first 5 weekdays, thereafter as required by Contractor for system optimization, if correlation established	Total organics using in-line Continuous Analyzer, Vapor flow rate	*total organics*	Total Organics- Field Series 8800 Continuous Analyzer	--	--	--	--
Soil Vapor- All Trench Segments Combined	SVE System- Treatment Building at manifold samling port	Daily for first 5 weekdays	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor - Individual trench segments	SVE well head at trench segment	Following restart for confirmaton of shut down criteria	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	--
Soil Vapor- *Ex-situ* Treatment Cell, Combined	SVE System- Treatment Building	Minimum of one sample per month during operation of SVE for *ex-situ* treatment cell and following restart spike tests to confirm shutdown	-	SVOCs, VOCs[1]	VOCs and SVOCs by EPA Method TO-15, except phenols by EPA Method TO-13A	--	--	1/10 (phenol)	
Excess Soil Excavated from *Augmented SVE Trenches*	Soil stockpiles from trench *excavation*	One soil sample per augmented *SVE trench*	PID	VOCs, SVOCs, Inorganics, and PCBs by SPLP and non-*leachate analysis of VOCs and* SVOCs[2]	SW-846 Methods 8260B, 8270C, 6010, and 8082	--	--	--	--
Water (biopolymer slurry after enzyme addition)	Biopolymer Slurry from SVE trench segments	*One-time*	Field tests as required to *confirm biopolymer slurry* breakdown	BOD, viscosity, and/or other *parameters to confirm* biopolymer slurry breakdown	SW-846 Method 405.1 for BOD; appropriate physical test *methods for viscosity or other* parameters to confirm biopolymer slurry breakdown	--	--	--	--
Subsurface Water- single combined water sample[3]	PRGS Pipe Collection Manhole	One sample following two successful Restart Spike Tests and one sample following each 90 days of additional SVE system operation	pH, specific conductance, temperature *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	--

TABLE E-1

Sampling Schedule and Analytical Parameters
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix Sampled	Sampling Area	Frequency	Field Parameters	Laboratory Analyses	Laboratory Methods	Duplicates	Trip Blanks	Field Equipment Blanks	MS/MSD
Subsurface water within each trench segment	Augmented SVE Trench Dewatering Wells	Semi-annual sampling of each operating trench segment during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods[5]	pH, specific conductance, temperature, water level *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Treated Subsurface Water/Effluent	PRGS Treatment Vessel-Effluent Sampling Port	Annual sampling after completion of the 2-year of Phase II(a) monitoring	pH, specific conductance, temperature *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Subsurface Water	Sand and Gravel Monitoring Wells S-1, S-4B and S-5	Semi-annual sampling during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods	pH, specific conductance, temperature, water level *(field filter samples for metals and PCBs)*	VOCs, SVOCs[4], some metals[4], cyanide, PCBs	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	1/10	1/20
Surface Water	Unnamed Ditch, at Locations SW-1, NSL-1 and SW-2	Semi-annual sampling during operation of the SVE system; quarterly sampling during the 1-year Phase I and 2-year Phase II(a) monitoring periods	Stream Observations	VOCs, SVOCs[6], some metals, cyanide	SW-846 Methods 8260B, 8270C, 6010, ILM04.1	1/10	1/ shipment	--	1/20
---	Thin Barrier Curtain Wall Piezometers	Quarterly during the 1-year Phase I and 2-year Phase II(a) monitoring periods	Water Level Measurements	--	--	--	--	--	--
Augmented SVE System	Wastewater Discharge Monitoring- Tank T-4 or discharge port	Prior to each batch discharge	--	VOCs with Approved Effluent Limits (see Attachment C-1 of FSP Addendum)	SW-846 Method 8260B	1/10	--	1/10	N/A

Key: VOCs= volatile organic compounds; SVOCs= semivolatile organic compounds; PCBs= polychlorinated biphenyls
SVE= soil vapor extraction
PID = Photoionization Detector
MS/MSD = Matrix Spike/Matrix Spike Duplicate
-- = None/Not Applicable
TBD = To be determined

[1] VOCs and SVOCs listed in Table 3-1 with Soil Vapor Standards. (Table 3-1 in the Design Report for the Attachment Z-1 Remedy)

[2] Analysis of VOCs and SVOCs with Site Specific Soil Exposure Concentrations in Table 2-2 of the Design Report for the Attachment Z-1 Remedy; additional analysis may be added if disposed offsite.

[3] If no water in PRGS system at end of Active Phase, sampling will be considered complete.

[4] VOCs and SVOCs listed in Table 2-1 with Acceptable Stream Concentrations.

[5] Additional samples may be collected during SVE system operation at the discretion of the ECC Site Trust.

[6] VOCs and SVOCs listed in Table 2-1 with Acceptable Surface Water Concentrations (Table 2-1 in the Design Report for the Attachment Z-1 Remedy)

TABLE E-2
Project Detection Limits and Reporting Limits -Soils
Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/Kg	Reporting Limit µg/Kg
GCMS Volatiles	**8260B/5035 Soil** ***5 gm purge***	
1,1,1,2-Tetrachloroethane	0.32	5.0
1,1,1-Trichloro-2,2,2-trifluoroethane	0.77	5.0
1,1,1-Trichloroethane	0.23	5.0
1,1,2,2-Tetrachloroethane	0.44	5.0
1,1,2-trichloro-1,2,2-trifluoroethane	0.61	5.0
1,1,2-Trichloroethane	0.29	5.0
1,1-Dichloroethane	0.07	5.0
1,1-Dichloroethene	0.29	5.0
1,1-Dichloropropene	0.25	5.0
1,2,3-Trichlorobenzene	1.35	5.0
1,2,3-Trichloropropane	0.62	5.0
1,2,4-trichlorobenzene	1.32	5.0
1,2,4-Trimethyl benzene	1.19	5.0
1,2-Dibromo-3-chloropropane	1.48	5.0
1,2-Dibromoethane	0.40	5.0
1,2-Dichlorobenzene	0.99	5.0
1,2-Dichloroethane	0.30	5.0
1,2-Dichloropropane	0.34	5.0
1,3,5-Trimethyl benzene	1.23	5.0
1,3-Dichlorobenzene	1.00	5.0
1,3-Dichloropropane	0.23	5.0
1,4-Dichlorobenzene	1.05	5.0
1,4-Dioxane	38.26	250.0
2,2'-Dichloropropane	0.33	5.0
2-Butanone	1.66	12.5
2-Chlorotoluene	0.76	5.0
2-Hexanone	1.96	12.5
3-Chloropropene	0.33	5.0
4-Chlorotoluene	0.99	5.0
4-Methyl-2-pentanone	2.25	12.5
Acetone	4.07	12.5
Acetonitrile	0.35	5.0
Acrolein	6.64	50.0
Acrylonitrile	3.08	50.0
Benzene	0.17	5.0
Bromobenzene	0.60	5.0
Bromochloromethane	0.31	5.0
Bromodichloromethane	0.30	5.0
Bromoform	0.30	5.0
Bromomethane	0.60	5.0
Carbon disulfide	0.06	5.0
Carbon tetrachloride	0.31	5.0
Chlorobenzene	0.38	5.0

TABLE E-2
Project Detection Limits and Reporting Limits -Soils
Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/Kg	Reporting Limit µg/Kg
Chloroethane	0.69	5.0
Chloroform	0.16	5.0
Chloromethane	0.38	5.0
Chloroprene	0.22	5.0
cis-1,2-Dichloroethene	0.21	5.0
cis-1,3-Dichloropropene	0.10	5.0
Cyclohexane	1.24	5.0
Dibromochloromethane	0.20	5.0
Dibromomethane	0.32	5.0
Dichlorodifluoromethane	0.18	5.0
Ethylbenzene	0.85	5.0
Ethylmethacrylate	2.71	50.0
Hexachlorobutadiene	1.41	5.0
Iodomethane	0.26	5.0
Isobutyl alcohol	25.58	250.0
Isopropyl benzene	1.17	5.0
Isopropyl ether	0.33	5.0
m,p-Xylene	1.72	10.0
Methacrylonitrile	3.16	50.0
Methyl acetate	1.05	5.0
Methylcyclohexane	1.59	5.0
Methylene Chloride	0.19	5.0
Methylmethacrylate	2.60	50.0
Methyl-tert-butyl-ether	0.12	5.0
Naphthalene	0.94	5.0
n-Butyl benzene	1.61	5.0
n-Propyl benzene	1.36	5.0
o-Xylene	0.70	5.0
Pentachloroethane	NA	NA
p-Isopropyl toluene	1.43	5.0
Propionitrile	22.49	250.0
sec-butyl Benzene	1.48	5.0
Styrene	0.48	5.0
tert-butyl Benzene	1.28	5.0
Tetrachloroethene	0.77	5.0
Toluene	0.32	5.0
trans-1,2-Dichloroethene	0.25	5.0
trans-1,3-Dichloropropene	0.21	5.0
trans-1,4-Dichloro-2-butene	1.27	20.0
Trichloroethene	0.28	5.0
Trichlorofluoromethane	0.40	5.0
Vinyl acetate	0.34	5.0
Vinyl Chloride	0.38	5.0

TABLE E-2
Project Detection Limits and Reporting Limits -Soils
Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/Kg	Reporting Limit µg/Kg
GC PCBs	**3550B/8082 Soil**	
1016	11.78	31
1260	5.00	31
1242	11.22	21
1232	7.51	31
1221	20.27	42
1248	7.52	21
1254	2.00	21

Method detection limits (MDLs) and Reporting Limits (RLs) provided by Compuchem Laboratories (November 2006)
NA = Not Available

TABLE E-3
Project Detection Limits and Reporting Limits -Water
Volatile Organics, Semi-Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/L	Reporting Limit µg/L	MDL µg/L	Reporting Limit µg/L
GCMS Volatiles	**5030b/5035/82620B** *25 mL purge*			
1,1,1,2-Tetrachloroethane	0.04	0.5		
1,1,1-trichloro-2,2,2-trifluoroethane	0.08	0.5		
1,1,1-Trichloroethane	0.03	0.5		
1,1,2,2-Tetrachloroethane	0.05	0.5		
1,1,2-trichloro-1,2,2-trifluoroethane	0.14	0.5		
1,1,2-Trichloroethane	0.03	0.5		
1,1-Dichloroethane	0.07	0.5		
1,1-Dichloroethene	0.05	0.5		
1,1-Dichloropropene	0.04	0.5		
1,2,3-Trichlorobenzene	0.05	0.5		
1,2,3-Trichloropropane	0.10	0.5		
1,2,4-trichlorobenzene	0.05	0.5		
1,2,4-Trimethylbenzene	0.03	0.5		
1,2-Dibromo-3-chloropropane	0.20	0.5		
1,2-Dibromoethane	0.06	0.5		
1,2-Dichlorobenzene	0.03	0.5		
1,2-Dichloroethane	0.06	0.5		
1,2-Dichloropropane	0.04	0.5		
1,3,5-Trimethylbenzene	0.03	0.5		
1,3-Dichlorobenzene	0.04	0.5		
1,3-Dichloropropane	0.06	0.5		
1,4-Dichlorobenzene	0.04	0.5		
1,4-Dioxane	5.49	25.0		
2,2,-Dichloropropane	0.10	0.5		
2-Butanone	0.41	2.5		
2-chloroethyl vinyl ether	0.85	5.0		
2-Chlorotoluene	0.03	0.5		
2-Hexanone	0.55	2.5		
3-Chloropropene	0.05	0.5		
4-Chlorotoluene	0.04	0.5		
4-Methyl-2-pentanone	0.40	2.5		
Acetone	0.38	2.5		
Acetonitrile	0.04	0.5		
Acrolein	1.59	5.0		
Acrylonitrile	0.50	5.0		
Benzene	0.02	0.5		
Bromobenzene	0.03	0.5		
Bromochloromethane	0.05	0.5		
Bromodichloromethane	0.06	0.5		
Bromoform	0.04	0.5		
Bromomethane	0.05	0.5		
Carbon disulfide	0.03	0.5		
Carbon tetrachloride	0.07	0.5		

TABLE E-3
Project Detection Limits and Reporting Limits -Water
Volatile Organics, Semi-Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/L	Reporting Limit µg/L	MDL µg/L	Reporting Limit µg/L
Chlorobenzene	0.02	0.5		
Chloroethane	0.11	0.5		
Chloroform	0.03	0.5		
Chloromethane	0.04	0.5		
Chloroprene	0.04	0.5		
cis-1,2-Dichloroethene	0.04	0.5		
cis-1,3-Dichloropropene	0.02	0.5		
Cyclohexane	0.05	0.5		
Dibromochloromethane	0.03	0.5		
Dibromomethane	0.04	0.5		
Dichlorodifluoromethane	0.05	0.5		
Ethylbenzene	0.04	0.5		
Ethylmethacrylate	0.33	5.0		
Hexachlorobutadiene	0.07	0.5		
Iodomethane	0.04	0.5		
Isobutyl alcohol	7.28	25.0		
Isopropyl benzene	0.04	0.5		
Isopropyl ether	0.11	0.5		
m,p-Xylene	0.04	1.0		
Methacrylonitrile	0.68	5.0		
Methyl acetate	0.13	0.5		
Methylcyclohexane	0.07	0.5		
Methylene chloride	0.06	0.5		
Methylmethacrylate	0.53	5.0		
Methyl-tert-butyl-ether	0.05	0.5		
Naphthalene	0.06	0.5		
n-Butyl benzene	0.05	0.5		
n-Propyl benzene	0.03	0.5		
o-Xylene	0.04	0.5		
p-Isopropyl toluene	0.07	0.5		
Propionitrile	3.31	25.0		
sec-Butyl Benzene	0.04	0.5		
Styrene	0.03	0.5		
tert-butyl benzene	0.05	0.5		
Tetrachloroethene	0.09	0.5		
Toluene	0.04	0.5		
trans-1,2-Dichloroethene	0.02	0.5		
trans-1,3-Dichloropropene	0.03	0.5		
trans-1,4-dichloro-2-butene	2.00	20.0		
Trichloroethene	0.04	0.5		
Trichlorofluoromethane	0.05	0.5		
Vinyl acetate	0.19	1.0		
Vinyl Chloride	0.06	0.5		
Trichlorofluoromethane	0.40	5.0		
Vinyl acetate	0.34	5.0		
Vinyl Chloride	0.38	5.0		

TABLE E-3
Project Detection Limits and Reporting Limits -Water
Volatile Organics, Semi-Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/L	Reporting Limit µg/L	MDL µg/L	Reporting Limit µg/L
GCMS Semi-Volatiles	**8270C Water** *3510C/8270C*		*3550B/8270C*	
1,1'-Biphenyl	2.00	10	49.54	330
1,2,4-Trichlorobenzene	2.91	10	39.20	330
1,2-Diphenylhydrazine	0.38	10	230.24	330
1,3-Dichlorobenzene	7.73	10	328.73	330
1,4-Dichlorobenzene	7.95	10	222.39	330
1-Methylnaphthalene	7.21	10	214.96	330
2,2'-oxybis(1-Chloropropane)	1.48	10	259.72	330
2,4,5-Trichlorophenol	1.55	10	31.73	330
2,4,6-Trichlorophenol	1.32	10	54.43	330
1,2-Dichlorobenzene	7.97	10	38.10	330
2,4-Dichlorophenol	1.45	10	38.02	330
2,4-Dimethylphenol	1.77	10	29.22	330
2,4-Dinitrophenol	5.30	20	78.80	330
2,4-Dinitrotoluene	1.33	10	552.27	660
2,6-Dinitrotoluene	1.28	10	52.08	330
2-Chloronaphthalene	1.93	10	56.72	330
2-Chlorophenol	1.71	10	44.89	330
2-Methylnaphthalene	2.33	10	31.95	330
2-Methylphenol	1.31	10	39.62	330
2-Nitroaniline	1.45	20	47.50	660
2-Nitrophenol	2.03	10	30.58	330
3,3'-Dichlorobenzidine	1.27	10	24.99	330
3/4-Methylphenol	2.77	20	92.35	660
3-Nitroaniline	1.75	20	33.69	660
4,6-Dinitro-2-methylphenol	1.58	20	36.70	660
4-Bromophenyl-phenylether	1.48	10	57.84	330
4-Chloro-3-methylphenol	1.87	10	56.05	330
4-Chloroaniline	1.69	10	66.35	330
4-Chlorophenyl-phenylether	1.65	10	44.54	330
4-Nitroaniline	2.04	20	31.82	660
4-Nitrophenol	0.92	20	54.52	660
Acenaphthene	1.62	10	42.19	330
Acenaphthylene	1.73	10	47.96	330
Acetophenone	1.71	10	136.32	330
Anthracene	1.15	10	47.18	330
Atrazine	0.86	10	35.87	330
Benzaldehyde	2.00	10	68.59	330
Benzo(a)anthracene	1.33	10	42.02	330
Benzo(a)pyrene	1.27	10	35.90	330
Benzo(b)fluoranthene	1.17	10	36.63	330
Benzo(g,h,i)perylene	1.41	10	26.62	330
Benzo(k)fluoranthene	1.92	10	61.99	330
Benzyl alcohol	2.76	10	80.11	330

TABLE E-3
Project Detection Limits and Reporting Limits -Water
Volatile Organics, Semi-Volatile Organics and PCB's
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	MDL µg/L	Reporting Limit µg/L	MDL µg/L	Reporting Limit µg/L
Bis(2-chloroethoxy)methane	1.92	10	43.68	330
Bis(2-chloroethyl)ether	1.82	10	33.26	330
bis(2-ethylhexyl)Phthalate	2.00	10	62.37	330
Butylbenzylphthalate	1.29	10	47.96	330
Caprolactam	1.02	10	34.54	330
Carbazole	1.54	10	44.79	330
Chrysene	1.40	10	47.72	330
Dibenzo(a,h)anthracene	1.39	10	34.84	330
Dibenzofuran	1.58	10	45.03	330
Diethylphthalate	1.44	10	47.65	330
Dimethylphthalate	1.32	10	45.55	330
Di-n-butylphthalate	1.51	10	51.20	330
Di-n-octylphthalate	1.28	10	60.76	330
Fluoranthene	1.43	10	43.92	330
Fluorene	1.29	10	46.66	330
Hexachlorobenzene	1.14	10	53.94	330
Hexachlorobutadiene	3.90	10	30.31	330
Hexachlorocyclopentadiene	9.46	10	75.94	330
Hexachloroethane	2.55	10	20.93	330
Indeno(1,2,3-cd)pyrene	1.21	10	26.87	330
Isophorone	1.98	10	39.50	330
Naphthalene	2.51	10	33.44	330
Nitrobenzene	2.12	10	43.00	330
N-Nitrosodimethylamine	1.56	10	44.62	330
N-Nitroso-di-N-propylamine	1.75	10	69.45	330
N-Nitrosodiphenylamine	2.42	10	75.89	330
Pentachlorophenol	5.41	20	30.91	660
Phenanthrene	1.48	10	46.10	330
Phenol	0.81	10	70.24	330
Pyrene	1.54	10	44.79	330
Pyridine	3.37	10	151.92	330
GC PCBs	**3510C/8082/608**			
Aroclor				
1016	0.358	0.93		
1260	0.277	0.93		
1242	0.317	0.625		
1232	0.578	0.93		
1221	1.608	1.25		
1248	0.422	0.625		
1254	0.155	0.625		

Method detection limits (MDLs) and Reporting Limits (RLs) provided by Compuchem Laboratories (November 2006)

TABLE E-4
Project Detection Limits and Reporting Limits -Soil and Water
Inorganics
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	IDL (P3) µg/L	IDL (P4) µg/L	Reporting Limit µg/L	Reporting Limit mg/Kg
ICP-AES Metals				
Aluminum	19.970	19.699	200	20.0
Antimony	1.112	1.885	10	1.0
Arsenic	2.558	3.090	10	1.0
Barium	0.084	0.114	10	20.0
Beryllium	0.097	0.345	5	0.5
Cadmium	0.148	0.447	5	0.5
Calcium	18.720	3.031	5000	500.0
Chromium	0.357	0.947	5	1.0
Cobalt	0.352	1.121	5	0.5
Copper	1.080	0.832	5	0.5
Iron	7.496	11.456	100	10.0
Lead	1.517	1.341	3	0.3
Magnesium	2.567	3.808	5000	500.0
Manganese	0.075	0.357	10	1.0
Nickel	0.508	1.707	5	4.0
Potassium	8.303	3.142	5000	500.0
Selenium	2.492	2.437	5	0.5
Silver	0.443	0.459	5	0.5
Sodium	110.214	88.271	5000	500.0
Thallium	3.736	2.740	10	1.0
Vanadium	0.315	0.351	20	2.0
Zinc	0.767	0.536	20	2.0
Additional compounds				
Molybdenum	0.453	1.092	5	5.0
Tin	4.193	2.412	100	10.0
Titanium	5.277	0.347	40	4.0
Bismith	2.095	1.791	50	5.0
Boron	1.405	N/A	200	50.0
Mercury				
Mercury	0.040		0.20	0.20
Spectrophotometric Cyanide				
Cyanide	1.20		10	10

Instrument detection limits (IDLs) and Reporting Limits (RLs) provided by Compuchem Laboratories (November 2006)

TABLE E-5
Project Reporting Limits - Soil Vapor
Volatile Organics and Semi-Volatile Organics
Environ-Chem Superfund Site
Zionsville, Indiana

Compound Name	Reporting Limit ppbv
Project VOCs and SVOCs	**TO14A/TO15**
1,1,1,2-Tetrachloroethane	0.5
1,1,2-trichloro-1,2,2-trifluoroethane	0.5
1,1-Dichloroethane	0.5
1,1-Dichloroethene	0.5
1,2,4-trichlorobenzene	2.0
1,2,4-Trimethylbenzene	0.5
1,2-Dibromoethane	0.5
1,2-Dichlorobenzene	0.5
1,2-Dichloroethane	0.5
1,2-Dichloropropane	0.5
1,3,5-Trimethylbenzene	0.5
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.5
2-Butanone	0.5
2-Hexanone	2.0
4-Methyl-2-pentanone	0.5
Acetone	2.0
Benzene	0.5
Bromodichloromethane	0.5
Bromoform	0.5
Bromomethane	0.5
Carbon disulfide	0.5
Carbon tetrachloride	0.5
Chlorobenzene	0.5
Chloroethane	0.5
Chloroform	0.5
Chloromethane	0.5
cis-1,2-Dichloroethene	0.5
cis-1,3-Dichloropropene	0.5
Dibromochloromethane	0.5
Ethylbenzene	0.5
Hexachlorobutadiene	2.0
m,p-Xylene	0.5
Methylene chloride	0.5
Methyl-tert-butyl-ether	0.5
Propyl benzene	0.5
o-Xylene	0.5
Styrene	0.5
Tetrachloroethene	0.5
Toluene	0.5
trans-1,2-Dichloroethene	0.5
trans-1,3-Dichloropropene	0.5
Trichloroethene	0.5
Vinyl Chloride	0.5
1,2 Dichlorobenzene	0.5

Reporting Limits (RLs) provided by Air Toxics in TO14A/15 Quality Manual dated 07/2006

TABLE E-6

Sampling Containers, Preservation, and Holding Times
Enviro-Chem Superfund Site
Zionsville, Indiana

Matrix	Parameter	Container	Preservation	Hold Times	Sample Volume
Water	VOCs	Glass Vials	HCl to pH <2; Cool to 4 °C	14 days	3 x 40 mL
Water	SVOCs	Amber Glass	Cool to 4 °C	7 days to extractions; 40 days until analysis	2 X 1000 mL
Water	some metals and cyanide[1]	Plastic	NaOH	14 days	500 ml
Soil	VOCs (SPLP)	Glass	Cool to 4 °C	14 days to leach; 14 days from leach to analysis	4 oz.
Soil	VOCs	Glass Vials	Cool to 4 oC 2 with 5 mL sodium bisulfate, 1 with 5 mL methanol	14 days	3 x 40 mL
Soil	SVOCs	Clear Glass	Cool to 4 °C	14 days to extractions 40 days until analysis	8 oz.
Soil	SVOCs, PCBs (SPLP)	Clear Glass	Cool to 4 °C	14 days to leach; 7 days from leach to analysis	8 oz.
Soil	Metals (not including mercury) (SPLP)	Clear Glass	Cool to 4 °C	180 days to leach; 180 days from leach to analysis	8 oz.
Soil Vapor	VOCs, 1,2-Dichlorobenzene	SUMMA Canister or Tedlar Bag	NA	7 days	1000 mL or 6000 mL
Soil Vapor	Phenol	XAD sorbent tubes	Cool to 4 °C	7 days	XAD sorbent tubes

Key:

VOCs = Volatile organic compounds
SVOCs = Semivolatile organic compounds
HCl = Hydrochloric acid
PCBs = Polychlorinated biphenyls
SPLP = Synthic Precipitation Leaching Procedure
Note 1: Analysis list from Table 2-1 of the Design Report for the Attachment Z-1 Remedy, Acceptable Stream Concentration list.

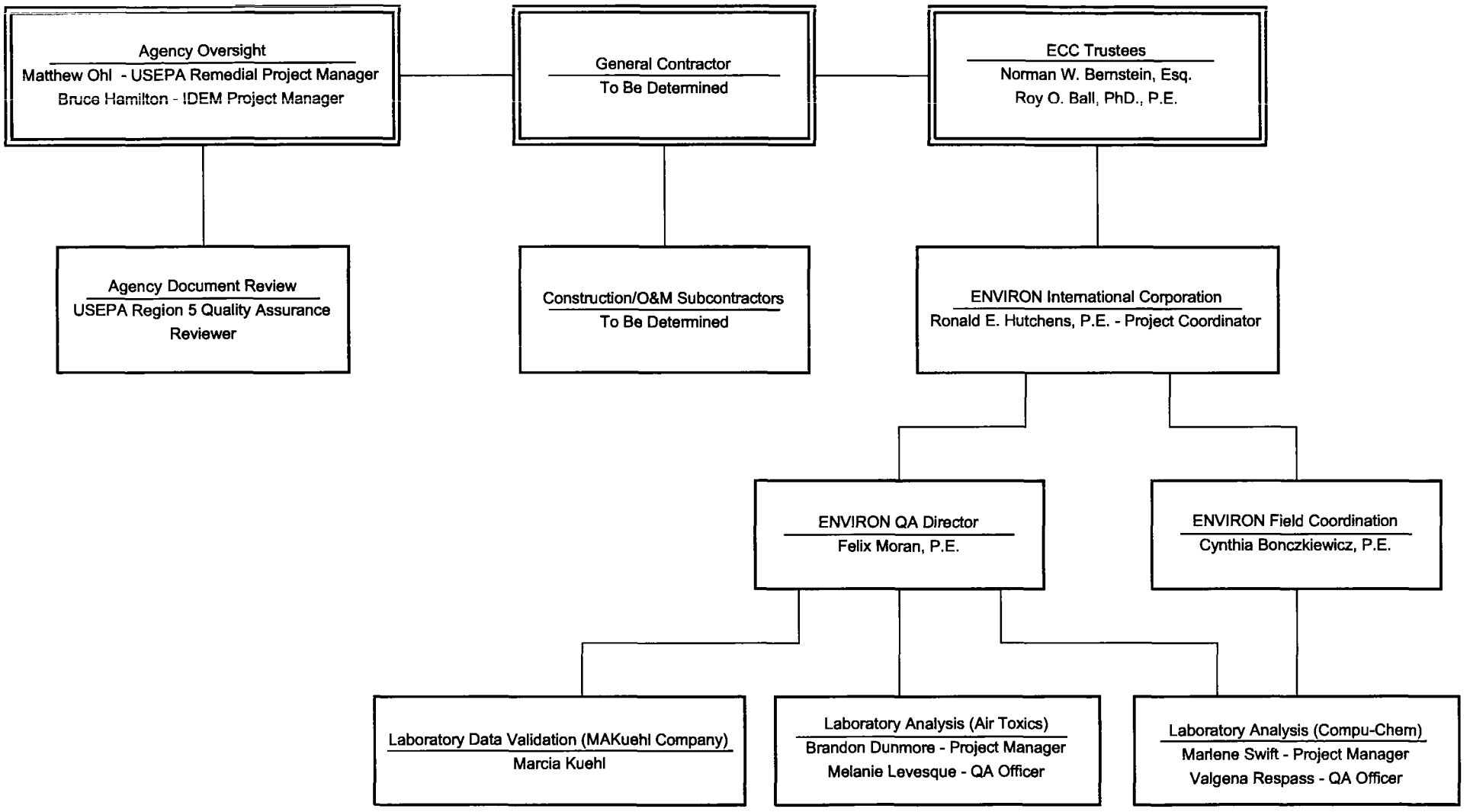
TABLE E-7

QA Objectives for Field Measurements
Enviro-Chem Superfund Site
Zionsville, Indiana

PARAMETER	METHOD[1] REFERENCE	PRECISION[2]	ACCURACY[3]	COMPLETENESS
WATER				
Standing Water Levels	Solinist or Keck Water Level Indicator	±0.01 ft.	±0.005 ft.	95%
Temperature	Electronic Temperature Probe (Horiba U-U-20/U-22)	±0.5°C	±1.0°C	95%
Conductivity	E120.1, Electrometric (Horiba U-20/U-22)	±25	±10 umho/cm^2	95%
pH	E150.1(Horiba U-22 probe)	±0.1 pH units	± 0.05 pH units	95%
SOIL				
Organic Vapor Screening	Photoionization Detector (PID)	±0.1-1.0 ppmV	±2.0 ppmV or 10% of reading	95%

NOTES:
1. Methods: E - *Method for Chemical Analysis for Water and Wastes* (U.S. EPA, 1983).
   SW - *Test for the Evaluation of Solid Waste*, SW-846, U.S. EPA, September 1986, Update III, June 1997.
   SM - *Standard Methods for Examination of the Water and Wastewater*, 18th ed. (APHA, 1992).
   ASTM - *Annual Book of ASTM Standards*, American Society of Testing and Materials, 1995.
2. Expressed as the acceptable deviation from the Scale.
3. Expected based on equipment manufacturer specifications.

FIGURES



ENVIRON

PROJECT ORGANIZATIONAL CHART
ENVIRO-CHEM SUPERFUND SITE
985 S. U.S. HIGHWAY 421
ZIONSVILLE, INDIANA

Figure
E-1

Drafter:	APR	Date:	12/07/06	Contract Number:	21-6585K	Approved:	Revised:

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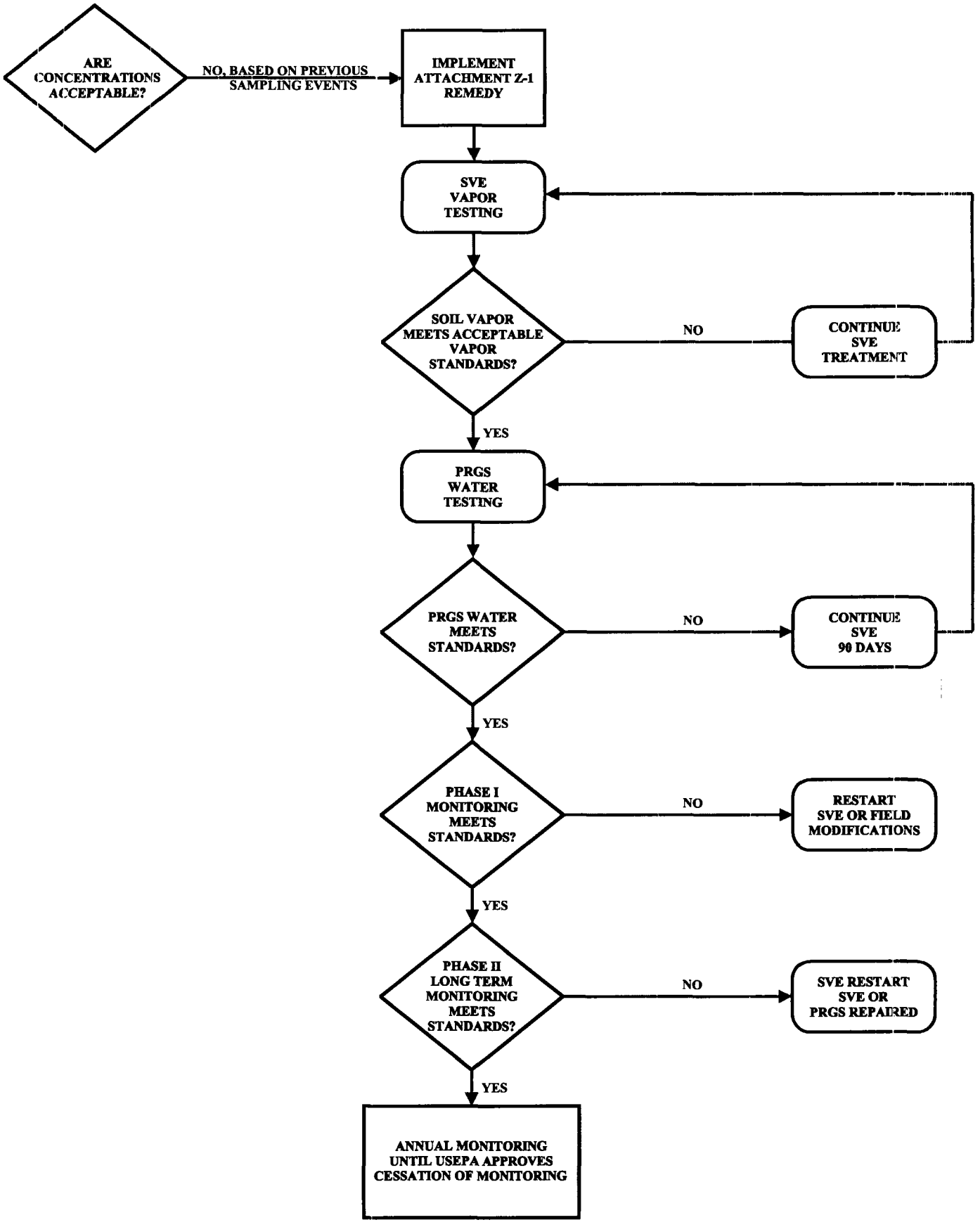

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ENVIRON

DECISION PERFORMANCE GOAL DIAGRAM
ENVIRO-CHEM SUPERFUND SITE
985 U.S. HIGHWAY 421
ZIONSVILLE, INDIANA

Figure
E-2

Drafter:	APR	Date: 12/07/06	Contract Number:	21-6585K	Approved:	Revised:

ATTACHMENT E-1

Standard Operating Procedures for Laboratory Analyses
Soil and Water
(on CD)

ATTACHMENT E-1

Standard Operating Procedures for Laboratory Analysis
Soil and Water

CONTENTS

ATTACHMENT E-1

Standard Operating Procedures for Laboratory Analysis
Soil and Water

C O N T E N T S

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: Hazardous Waste Management SOP Section #: 12.1 Revision #: 7

SOP Title: Hazardous Waste Disposal

Effective date: (QA fills in) 2/2/04

- Procedure prepared by: [signature] Date: 1/28/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane Ellmore Date: 2/2/04
- Reason for change: Remove CH_2Cl_2 references
- This procedure meets the requirements of the following approved method references: RCRA 40 CFR (261-271); OSHA 29 CFR (1910.120 & 1910.1200); HMTA 40 CFR (171-180; HM-181 & HM126F); CWA 40CFR (403.5); SARA 40 CFR (355, 370); 15A NCAC 13A; Dept of Ag. 7CFR (301.81)

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

[signature]

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] Date: 2/11/05

Annual Review—Signature: [signature] Date: 5/3/06

Annual Review—Signature: _____ Date: _____

Hazardous Waste Management & Safety SOP 12.1: Hazardous Waste Disposal

Table of Contents

1.0 Scope and Application

This SOP provides guidelines for the safe and legal collection, storage, and disposal of all wastes generated by the laboratory's operations. These guidelines are applicable to all employees and subcontractors employed to handle samples, standards, or laboratory chemicals.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

All samples received and all waste generated by the laboratory in sample processing must be handled and disposed of in a manner that is compliant with state and federal regulations. Procedures are outlined for the safe handling and disposition of waste comprising a variety of classifications or “streams”. Staff who manage the hazardous waste are identified and receive specific on-going training.

3.0 Definitions

3.1 Hazardous Waste Technician - The hazardous waste technician is responsible for the following duties:

3.1.1 Handles all laboratory wastes streams as described in section 7.0 of this SOP.

3.1.2 Interacts with waste disposal companies to schedule waste removal.

3.1.3 Studies applicable ways to reduce the amount of hazardous waste generated.

3.1.4 Trains other employees in specific hazardous waste management when necessary.

3.1.5 Serves as an emergency coordinator.

3.2 Chemical Hygiene Officer - The Chemical Hygiene Officer is responsible for the following duties:

3.2.1 Coordinates training sessions and information exchange concerning chemical spills, waste handling, and the use of emergency equipment.

3.2.2 Investigates and reports accidents.

3.2.3 Develops emergency plans and spill response.

3.2.4 Reviews waste handling and disposal procedures.

3.2.5 Serves as an emergency coordinator.

3.3 Sample Custodians

3.3.1 The Sample Custodians dispose of expired samples in the appropriate drum in their satellite area.

3.4 Laboratory Supervisors

3.4.1 Laboratory Supervisors are responsible for implementing these guidelines.

3.5 Laboratory Staff

3.5.1 Laboratory Staff are responsible for following these guidelines. Waste minimization efforts by laboratory staff are also encouraged.

3.6 Emergency Coordinators

3.6.1 Detailed responsibilities of Emergency Coordinators are outlined in the CompuChem Contingency Plan.

3.7 Waste Minimization Coordinator

3.7.1 The Waste Minimization Coordinator maintains and oversees the company's Waste Minimization Plan. The Hazardous Waste Technician currently handles the responsibilities of this position.

3.8 Waste Streams - The following list describes each type of waste stream generated at CompuChem.

3.8.1 Waste Methylene Chloride

3.8.1.1 Used as a solvent in various extractions and to clean glassware

3.8.2 Waste Freon 113

3.8.2.1 Used in Total Petroleum Hydrocarbon extractions

3.8.3 Waste Mixed Flammable Solvents

3.8.3.1 Used in extractions

3.8.4 Plants Scraps

3.8.4.1 Solid wastes, such as filters, left from laboratory processes

3.8.5 Flammable Solvents in Vials

3.8.5.1 Very small or unopenable vials produced by GC, GC/MS, and other labs

3.8.6 019 Waste

3.8.6.1 Acidic extraction waters, purged water samples, and waste mixed acids that don't meet RCRA metal regulations, contain solvents or are otherwise contaminated, and are ineligible for disposal to the sewer system

3.8.7 Waste Mixed Acids

3.8.7.1 Cyanide distillations, ICP, AA instruments, digestates, and glassware acid bath

3.8.8 Waste Sodium Hydroxide and Cyanide

3.8.8.1 Cyanide distillations

3.8.9 Purged Soil Samples

3.8.9.1 Expired solid samples from the sample cooler

3.8.10 Empty Sample Containers

3.8.10.1 A non-hazardous waste stream from the purged water samples and other sources

3.8.11 Broken Glassware

3.8.11.1 A non-hazardous waste stream that comes from all laboratory processes and glassware prep.

3.8.12 Expired Chemicals

3.8.12.1 Expired reagents and standards from all labs

3.8.13 Purged Water Samples

3.8.13.1 Expired water samples from the sample cooler

3.8.14 Other

3.8.14.1 The Chemical Hygiene Officer and the Hazardous Waste Technician will specify other waste streams as needed.

3.9 Waste Disposal Companies

3.9.1 To prevent improper disposal, the licensed companies that dispose of this laboratory's hazardous waste perform additional testing before disposal to determine the contents of the shipment containers.

3.10 RCRA – Resource Conservation and Recovery Act

3.11 DOT – Department of Transportation

4.0 Safety

4.1 Emergency Procedures

4.1.1 Contingency Plan

4.1.1.1 All emergency procedures may be found in the facility's Contingency Plan.

4.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

4.3 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment & Supplies

5.1 Moving Equipment

5.1.1 The solvent storage/waste staging room is stocked with equipment, such as handtrucks and a drum cart, to help in the movement of containers of waste.

5.2 Waste Compactor

5.2.1 An electric compactor is located outside and is used to compact the purged soil sample and empty sample container waste streams.

6.0 Sample Collection, Preservation, & Storage

N/A

7.0 Quality Control

7.1 Disposal Arrangements

7.1.1 All waste is handled in compliance with all applicable local, state, and federal laws. A licensed waste disposal company (or companies) is contracted to dispose of hazardous wastes.

7.2 Compliance with Federal Regulations

7.2.1 The laboratory is required to comply with applicable parts of the regulations listed in Section 11.0 of this SOP.

7.3 All personnel receive training by the Human Resources department covering "Right to Know", the Chemical Hygiene Plan, OSHA Standards, and the Contingency Plan/Emergency Action Plan.

7.4 Employees identified as hazardous waste handlers or managers receive additional, more specific training including hazardous waste management, RCRA and DOT regulations, and managing hazardous waste and used oil, for example. Continued training is required to be received on an annual basis and may be administered

internally by trained laboratory staff or through an external provider. An example of the training checklist used for internal training is provided as Attachment 1. Certificates and other documentation are maintained in the employee training files.

7.5 Logbook Review

7.5.1 All logbooks are reviewed by the manager and audited on a periodic basis by the Quality Assurance department.

8.0 Calibration & Standardization

N/A

9.0 Procedure

9.1 Documentation Requirements

Documentation must follow the requirements in QC SOP: Proper Documentation Procedure. Documentation requirements include the following.

9.1.1 Hazardous Waste Manifest

9.1.1.1 A North Carolina Hazardous Waste Manifest (Attachment 2) is completed and accompanies all shipments to the licensed hazardous waste contractor. The manifest is signed by the generator of the hazardous waste and the transporter to acknowledge receipt of the materials. When the materials have been disposed of, a completed copy of the manifest with signatures from the disposal facility is returned to the laboratory. The Hazardous Waste Technician maintains a file of these records.

9.1.2 Sample Accident Report (Attachment 3) and Spill Report (Attachment 4)

9.1.2.1 The Sample Accident Report and Spill Report must be completed for each event and are administered by the Chemical Hygiene Officer.

9.1.2.2 Spills are handled according to the guidelines in Hazardous Waste Management and Safety SOP 12.2, “Spill Control and Cleanup”.

9.1.3 Inspection Logs

9.1.3.1 The following logs must be maintained:

9.1.3.1.1 Outside 90-day Storage Area Daily Inspection. Logbook 18D (Attachment 5).

9.1.3.1.2 Inside 90-day Storage Area Daily Inspection Logbook.

9.1.3.1.3 Manifest Tracking Logbook 18E (Attachment 6).

9.2 Disposal by Waste Stream

The procedure for handling each kind of waste stream is described below. Grounding procedures are described and diagrammed in Attachment 7 and Attachment 8.

9.2.**1** Waste Freon 113

9.2.1.1 Disposal Container

9.2.1.1.1 Use a DOT-approved steel 55-gallon closed-head drum for shipment.

9.2.1.2 Personal Protective Equipment

9.2.1.2.1 Wear a lab coat, full-face respirator (or half-face respirator with safety glasses or face shield) and appropriate gloves.

9.2.1.3 Method

9.2.1.3.1 Transfer the Freon 113 into the satellite container by either pouring or pumping. Fill to within about two inches of the top of the drum.

9.2.1.4 Storage Location and Labeling

9.2.1.4.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.2 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.**2** Waste Mixed Flammable Solvents

9.2.**2**.1 Special Instructions

9.2.**2**.1.1 Use grounding method #1 when pouring from mixed flammable solvent satellite containers into the 55-gallon drum..

9.2.2.2 Disposal Container

9.2.2.2.1 Use a DOT-approved steel 55-gallon closed-head drum for shipment.

9.2.2.3 Personal Protective Equipment

9.2.2.3.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.2.4 Method

9.2.2.4.1 Transfer the solvents from the satellite container into the drum by pouring. Fill to within about two inches of the top of the drum.

9.2.2.5 Storage Location and Labeling

9.2.2.5.1 The filled container should be taken to the outside storage area and be labeled as directed in Section 9.3.3 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.**3** Plant Scraps

9.2.3.1 Disposal Container

9.2.3.2.1 Use a DOT-approved 55-gallon open-head steel drum or plastic drum.

9.2.3.2 Personal Protective Equipment

9.2.3.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.3.3 Method

9.2.3.3.1 Transfer the plant scraps from the satellite container into the steel drum. Use the inner liner of a satellite container or some other flat object to compact the plant scraps so that as much waste as possible may be placed in a container. Compacting is optional.

9.2.3.4 Storage Location and Labeling

9.2.3.4.1 Filled containers should be taken to the outside storage area and be labeled as directed in Section 9.3.4 of this SOP.

9.2.**4** Flammable Solvent in Vials

9.2.4.1 Disposal Container

9.2.4.1.1 Transfer the vials from the satellite container into a DOT-approved 55-gallon open-head steel drum.

9.2.4.2 Personal Protective Equipment

9.2.4.2.1 Wear a lab coat, safety glasses, and appropriate gloves.

9.2.4.3 Storage Location and Labeling

9.2.4.3.1 Filled containers should remain in the solvent storage/waste staging room and be labeled as directed in Section 9.3.5 of this SOP.

9.2.**5** 019 Waste

9.2.5.1 Disposal and Storage Containers

9.2.5.1.1 Transfer 019 waste from satellite containers into a DOT-approved steel 55-gallon closed-head drum or plastic drum.

9.2.5.2 Purged Water Samples

9.2.5.2.1 Transfer liquid samples from cooler. Empty the sample bottles by purging directly into a DOT-approved 55-gallon closed-head drum. Dispose of the empty sample container according to section 9.2.10.

9.2.5.3 Personal Protective Equipment

9.2.5.3.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.5.4 Storage Location and Labeling

9.2.5.4.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.6 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.**6** Waste Mixed Acids

9.2.6.1 Storage Containers

9.2.6.1.1 Transfer waste mixed acids from satellite containers into a 55-gallon non-metal, acid-resistant drum for storage.

9.2.6.2 Personal Protective Equipment

9.2.6.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be worn.

9.2.6.3 Storage Location and Labeling

9.2.6.3.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.7 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.**7** Waste Sodium Hydroxide and Cyanide

9.2.7.1 Storage Container

9.2.7.1.1 Transfer the waste sodium hydroxide and cyanide from satellite containers to a 55-gallon non-metal, corrosion-resistant drum for storage.

9.2.7.2 Personal Protective Equipment

9.2.7.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be worn.

9.2.7.3 Storage Location and Labeling

9.2.7.3.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.8 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.**8** Purged Soil Samples

9.2.8.1 Disposal Container

9.2.8.1.1 Place purged soil samples with containers into a DOT-approved 55-gallon, open-head steel drum.

Note: The identifying labels, including client or project names and other information, must be removed prior to disposal.

9.2.8.2 Personal Protective Equipment

9.2.8.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A face shield may also be worn.

9.2.8.3 Storage Location and Labeling

9.2.8.3.1 Filled containers should be moved to the outside storage area and be labeled as directed in Section 9.3.9 of this SOP.

9.2.8.4 Foreign Soil and Domestic Quarantine Soil Samples

9.2.8.4.1 All samples arriving from outside the continental United States and from quarantined areas of the continental United States, are to be heated to 500 °F

for two minutes prior to being disposed in the manner that domestic soils are disposed. These samples are identified by the presence of red- and white-striped tape on the sample container.

- Option 1: Place the soil samples with container on a metal tray in the muffle furnace in the Sample Preparation Laboratory. Heat to 500°F for two full minutes. Dispose of the cooled samples in accordance with this SOP.

- Option 2: Samples and sample containers may be placed in a 55 gallon, open-head steel drum marked "purged soil, foreign and domestic quarantine". This container can be shipped as is to a waste incineration facility that is permitted by the USDA for foreign and domestic soil disposal.

Note: This includes the filter and residue, if TCLP leaching is done.

9.2.**9** Empty Sample Containers

9.2.9.1 Storage Container

9.2.9.1.1 Empty and broken sample containers should have labels removed and are then disposed of in a commercial dumpster.

9.2.9.2 Personal Protective Equipment

9.2.9.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A face shield may also be worn.

9.2.1**0** Broken Glassware

9.2.10.1 Disposal Container

9.2.10.1.1 Broken glassware will be transferred from the satellite container directly to a dumpster.

9.2.10.2 Personal Protective Equipment

9.2.10.2.1 Wear a lab coat, safety glasses, and appropriate cut-proof gloves (Kevlar, for example). Also wear a pair of flexible gloves over the cut-proof gloves (latex, for example).

9.2.10.3 Method

9.2.10.3.1 Transfer the broken glassware container to the dumpster using the hand trucks. Transfer the container into the dumpster.

9.2.1**1** Expired Chemicals and Standards

9.2.1**1**.1 When a chemical reaches its shelf life, it must be disposed of as hazardous waste. These chemicals should be boxed and given to the Hazardous Waste Technician for disposal. The hazard class of each chemical should be provided to the Waste Technician with the chemicals so that the chemicals can be directed to the proper waste stream.

9.2.1**1**.2 Hazard Classes and Characteristics

- Non-Halogenated Flammables – Acids
- Halogenated Flammables – Bases
- Pesticides – Poisons
- Oxidizers – Reactives
- Inorganics (dry)

9.2.11.3 Disposal by Hazard Class

9.2.11.3.1 Non-Halogenated Flammable

- Add to the mixed flammable solvent waste stream as described above.

9.2.11.3.2 Halogenated Flammables

- Must be lab-packed or bulked in a 5-gallon DOT-approved steel pail.

9.2.11.3.3 Pesticides

- Must be lab-packed

9.2.11.3.4 Oxidizers

- Must be stored away from organic material

9.2.11.3.5 Inorganic Chemicals (dry)

- Stable chemicals can be lab-packed for incineration.

9.2.11.3.6 Acids

- If clean, acids can be directed to the waste mixed acid stream for disposal to the sewer as described above. Otherwise, they should be directed to the 019 waste stream.

9.2.11.3.7 Bases

- If clean, bases can be used to neutralize clean acid waste. Otherwise, they should be lab-packed for disposal by incineration.

9.2.11.3.8 Poisons

- Will be assessed for other characteristics and directed to the appropriate waste stream

9.2.11.3.9 Reactives

- If the chemical will react with water or air, or will produce a toxic gas when mixed with

another chemical, the material should be lab-packed for disposal by incineration.

9.2.1**2** Purged Water Samples

9.2.1**2**.1 Disposal Container

9.2.12.1.1 For amber liters, gallons and plastics, see Section 9.2.**9**.

9.2.12.1.2 For volatile samples, the container and sample are placed in a 55-gallon drum marked "**vials & acid**."

9.2.12.2 Personal Protective Equipment

9.2.12.2.1 Wear a lab coat, safety glasses, and appropriate gloves.

9.2.12.3 Method

9.2.12.3.1 The Sample Custodian is responsible for providing water samples to the Hazardous Waste Technician for disposal. The samples are bulked into the above-described container. Only clear samples are to be bulked. Unclear samples are to be diverted to the 019 water waste stream.

9.2.12.4 Storage Location and Labeling

9.2.12.4.1 Filled containers should be moved to the outside storage area and be labeled.

9.2.12.4.2 See Section 9.2.**6**.3 for Waste Mixed Acids.

9.2.13 Other Wastes

9.2.13.1 All other wastes will be evaluated by the Safety Officer, the Hazardous Waste Technician, and if necessary by the contracted disposal company for the appropriate means of disposal.

9.3 Labeling Requirements for Waste Streams

Examples of labels required for satellite accumulation containers and shipment containers can be found in Attachment 9 and Attachment 10.

9.3.1 Waste Freon 113

9.3.1.1 Satellite Accumulation containers must be labeled with the following information:

9.3.1.1.1 Hazardous Waste Label

9.3.1.1.2 Hazard Index:

- Health: 2
- Flammability: 1
- Reactivity: 0

9.3.**1**.2 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.**2** Waste Mixed Flammable Solvents

9.3.**2**.1 Satellite accumulation containers must be labeled with the following information:

9.3.3.1.1 Hazardous Waste Label

9.3.3.1.2 Hazard Index:

- Health: 3
- Flammability: 3
- Reactivity: 0
- Flammable Liquid Label

9.3.**2**.2 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.**3** Plant Scraps

9.3.**3**.1 Satellite accumulation containers must be labeled with the following information:

9.3.3.1.1 Hazardous Waste Label

9.3.3.1.2 Hazard Index:

- Health: 4
- Flammability: 3
- Reactivity: 0
- Cancer Hazard Label
- Flammable Liquid Label

9.3.**3**.2 Shipment containers must be labeled according to 49 CFR 172.101. See Attachment 9 for these requirements.

9.3.**4** Flammable Solvent in Vials

9.3.**4**.1 Satellite accumulation containers must be labeled with the following information:

9.3.4.1.1 Hazardous Waste Label

9.3.4.1.2 Hazard Index:

- Health: 2
- Flammability: 3
- Reactivity: 0
- Flammable Liquids Label

9.3.**4**.2 Shipment containers must be labeled according to 49 CFR 172.101.

9.3.**5** 019 Waste

9.3.**5**.1 Satellite Accumulation containers must be labeled with the following information:

9.3.5.1.1 Hazardous Waste Label with the words "019"

9.3.**5**.1.2 Hazard Index:

- Health: 3
- Flammability: 2
- Reactivity: 3
- Corrosive Label
- Oxidizer Label

9.3.**5**.1.3 Sample bottles that contain 019 waste are not required to be labeled.

9.3.**5**.2 Shipment containers must be labeled according to 49 CFR 172.101.

9.3.**6** Waste Mixed Acids **and Vials & Acid**

9.3.**6**.1 Satellite accumulation containers must be labeled with the following information:

9.3.6.1.1 Hazardous Waste Label

9.3.6.1.2 Hazard Index:

- Health: 3
- Flammability: 0
- Reactivity: 2
- Corrosive Label
- Oxidizer Label

9.3.**6**.2 Storage Containers must be labeled with the following information:

- Hazardous Waste Label b/w Non-hazardous Waste Label.
- Accumulation Start Date

9.3.**7** Waste Sodium Hydroxide with Cyanide

9.3.**7**.1 Satellite Accumulation containers must be labeled with the following information:

9.3.**7**.1.1 Hazardous Waste Label

9.3.7.1.2 Hazard Index:

- Health: 3
- Flammability: 0
- Reactivity: 3

9.3.7.1.3 Corrosive Label

9.3.**7**.2 Storage containers must be labeled with the following information:

9.3.7.2.1 Hazardous Waste Label b/w Non-hazardous waste label

9.3.7.2.2 Accumulation Start Date

9.3.**8** Purged Soil Samples

9.3.**8**.1 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.**9** Empty Sample Containers

9.3.**9**.1 These Shipment Containers do not have to be labeled. To avoid confusion and inspection, they may be labeled with a

"Non-regulated Waste" label. Do not fill in the information on this label.

9.3.1**0** Broken Glassware

9.3.1**0**.1 The satellite accumulation container does not have to be labeled. To avoid confusion and inspection, it may be labeled with a **"**Non-regulated Waste**"** label. Do not fill in the information on this label.

9.3.11 Expired Chemicals

9.3.11.1 All expired chemicals will be lab-packed and, therefore, labels will be used in accordance with the characteristics of the chemicals.

9.3.1**2** Other Wastes

9.3.1**2**.1 The Chemical Hygiene Officer and/or the Hazardous Waste Technician determine the appropriate labeling requirements.

9.4 Accumulation and Storage Requirements

9.4.1 Satellite Accumulation

9.4.1.1 Time Limitations

9.4.1.1.1 Waste may be accumulated for an indefinite time in satellite accumulation. When the 55 gallon drum is full, it is to be dated and moved to 90 day storage. Once the drum is dated, it must be moved to the 90 day storage within 3 days.

9.4.1.2 Volume Limitations

9.4.1.2.1 No more than 55 gallons of total waste from the same waste stream may be accumulated in satellite containers in one storage area. For example, a laboratory may store **flammable liquids** in a 55-gallon drum, or it may store many containers whose volume equals 55 gallons.

9.4.1.3 Labeling Limitations

9.4.1.3.1 Hazardous waste labels cannot be dated if they are in satellite accumulation. See Section 9.3 of this SOP for satellite accumulation labeling requirements.

9.4.2 Solvent Storage/Waste Staging Room

9.4.2.1 Time Limitations

9.4.2.1.1 Storage time is limited to 90 calendar days in this room.

9.4.2.2 Labeling Requirements

9.4.2.2.1 Accumulation Start Dates on hazardous waste labels must be filled in once the waste is stored in this room. See Section 9.3 of this SOP for solvent storage/waste staging room requirements.

9.4.3 Outside Storage Area

9.4.3.1 Time Limitations

9.4.3.1.1 Storage time is limited to 90 calendar days in this area.

9.4.3.2 Labeling Requirements

9.4.3.2.1 Accumulation Start Dates on hazardous waste labels must be filled in once the waste is stored in the outside storage area. See also section 9.3 of this SOP for outside storage labeling requirements.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, NaOH, Zn Acetate, or H_2SO_4 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab

11.0 References

11.1 Resource Conservation and Recovery Act (RCRA) (40 CFR 261-271)

11.2 Occupational Safety and Health Administration (OSHA) (29 CFR 1910.120 and 1910.1200)

11.3 Hazardous Materials Transportation Act (HMTA) (49 CFR 171-180: HM-181 and HM126F)

11.4 Clean Water Act (CWA) (40 CFR 403.5)

11.5 Superfund Amendments and Reauthorization Act (SARA) (40 CFR 355, 370)

11.6 NC Hazardous Waste Rules (15A NCAC 13A: same as RCRA)

11.7 Department of Agriculture (7CFR 301.81)

11.8 QCSOP: Proper Documentation Procedures

11.9 QCSOP: Numerical Data Reduction

11.10 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

11.11 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

11.12 NELAC Standards, **approved May 2001**, plus revisions

11.13 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

11.14 New York State Environmental Laboratory Approval Program, Certification Manual, June 2000.

11.15 CompuChem Quality Manual, Revision **4**, 12/1**0**/0**3**, plus revisions

11.16 Sample Control SOP 4.1, "Receiving Samples"

11.17 Sample Control SOP 4.6, “Storing Samples”

11.18 Hazardous Waste Management and Safety SOP 12.2, “Spill Control and Cleanup”

12.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

12.1 Attachment 1 – Training Checklist

12.2 Attachment 2 - NC Hazardous Waste Manifest

12.3 Attachment 3 – Sample Accident Report

12.4 Attachment 4 – Spill Report

12.5 Attachment 5 – Outside 90-day Storage Area Daily Inspections Log

12.6 Attachment 6 - Manifest Tracking Log

12.7 Attachment 7 - Schematic of Grounding Method 1

12.8 Attachment 8 - labels for satellite accumulation containers for waste streams

12.9 Attachment 9 – hazardous waste shipment container labels, including a table of DOT-required labels per waste stream

Attachment 1

CompuChem Laboratories

Specific Hazardous Waste Training Record

Employee name: _____ ID no.: _____

Job title: _____

I certify that I have received training in the following areas on the date(s) indicated, and that I understand all information presented.:

Employee Initials	Trainer Initials	Date	Area covered by training
_____	_____	_____	1. Specific hazardous wastes to be managed: _____ _____ _____
_____	_____	_____	2. Proper container(s) to be used
_____	_____	_____	3. Placement of waste into container(s)
_____	_____	_____	4. Required container labels
_____	_____	_____	5. Dating containers
_____	_____	_____	6. Satellite accumulation requirements (if applicable)
_____	_____	_____	7. Acid/base neutralizations
_____	_____	_____	8. Emergency notifications and response
_____	_____	_____	9. Container inspection procedures
_____	_____	_____	10. Lab safety manual hazardous waste policy review

Indicate subject areas in which the employee is not required to be trained by writing an "X" in the date column.

Employee Signature _____ Date _____

Trainer Signature _____ Date _____

Attachment 2

Please print or type. (Form designed for use on elite (12-pitch) typewriter.) Form Approved. OMB No. 2050-0039.

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. NCR000071606081 — Manifest Document No.
2. Page 1 of 1 — Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address: CompuChem Environmental Corporation, 501 Madison Ave., Cary, NC 27513
4. Generator's Phone (919) 379-4000

A. State Manifest Document Number
B. State Generator's ID
C. State Transporter's ID
D. Transporter's Phone (865)354-6827
E. State Transporter's ID
F. Transporter's Phone
G. State Facility's ID FLD980711071
H. Facility's Phone (352) 373-6066

5. Transporter 1 Company Name: T.A.G. Transport — 6. US EPA ID Number: TNR000003111
7. Transporter 2 Company Name — 8. US EPA ID Number
9. Designated Facility Name and Site Address: Perma-Fix of Florida, 1940 NW 67th Place, Gainesville, FL 32653 — 10. US EPA ID Number: FLD980711071

GENERATOR

11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	HM	12. Containers No.	12. Containers Type	13. Total Quantity	14. Unit Wt/Vol	Waste No.
a. Hazardous Waste, Liquid, n.o.s., 9, NA3082, PGIII (contains: methylene chloride, Hexane)	X	009	DF	04320	P	F002/F003
b. Waste Corrosive Liquid,Acidic,Inorganic,N.O.S.,8,UN3264, PGII,(Contains:Hydrochloric Acid,Nitric Acid)	X	001	DF	00398	P	D002
c. RQ,Waste Flammable Liquid,N.O.S.,3,UN1993,PGII (Contains:Methanol,Hexane)	X	003	DM	01350 THJ 00900	P	D001
d. Waste Corrosive Liquid,Acidic,Inorganic,N.O.S.,8,UN3264, PGII,(Contains:Hydrochloric Acid)	X	004	DF	01600	P	D002

J. Additional Descriptions for Materials Listed Above
11a. 019 Water, Profile#29891 D80 B204 M041
11b. Mixed Acids, Profile#32936-C - D73 B104 M141
11c. Mixed Flammables, Profile#32940 - D73 B204 M061
11d. Vials and Acid,Profile#32937-C D-D60 B104 M141

K. Handling Codes for Wastes Listed Above
501, M141
D-M141
A-M041 B-M141 -e M061

15. Special Handling Instructions and Additional Information
24 Hour Emergency # 865-376-0193
ERG# 11a.171 11b.154 11c.128 11d.154
RQ=D001>100 lbs

16. **GENERATOR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations.
If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; **OR**, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name: Carl A. Harker — Signature: Carl A. Harker — Month Day Year: 10/6/01

TRANSPORTER

17. Transporter 1 Acknowledgement of Receipt of Materials
Printed/Typed Name: Timothy H. Johnson — Signature: Timothy H. Johnson — Month Day Year: 10/6/01
18. Transporter 2 Acknowledgement of Receipt of Materials
Printed/Typed Name — Signature — Month Day Year

FACILITY

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.
Printed/Typed Name: Thomas A. Hamrick — Signature — Month Day Year: 10/8/01

Style CF 17 LABELMASTER, AN AMERICAN LABELMARK CO., CHICAGO, IL 60646 (800)621-5808 — EPA Form 8700-22 (Rev. 9-88) Previous editions are obsolete.

ORIGINAL-RETURN TO GENERATOR

PRINTED ON RECYCLED PAPER USING SOYBEAN INK — PRINTED WITH SOY INK

Attachment 3

CompuChem
a division of Liberty Analytical Corporation

SAMPLE ACCIDENT REPORT

Date & Time accident occurred: _____ / _____

Employee(s) involved: _____

Was anyone injured? ☐Yes ☐No Name: _____

If yes, an injury report must be filled out in the Human Resources Department

Where did accident occur? _____

What analyses were affected? _____

What was the cause of the accident? _____

_____

_____

_____

Sample ID: _____ CCN: _____

Case: _____ SDG: _____

Container size: _____ Bottle ___ of ___

CSR Notified: _____ Date: _____

accform1 – 7/3/01:dce

Attachment 4

Spill Report

CompuChem a Division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

Spill Information			
Date of spill	Time of spill	☐ a. m.	☐ p. m.
Material spilled			
Location of spill			
Brief description of spill:			
Quaintly spilled	Fire? ☐Yes ☐No	Explosion? ☐Yes ☐ No	
Intensity: ☐ Class A fires in ordinary combustible materials (wood, cloth, paper, rubber)			
☐ Class B fires in flammable liquids (oils, greases, tars, flammable gases)			
☐ Class C fires engendered by energized electrical equipment			
☐ Class D fires in combustible metals (Mg, Ti, Zr, Na, Li, K)			
Brief description of fire/explosion:			
Spill contained? ☐ Yes ☐ No	Area evacuated? ☐ Yes ☐ No		
Local authorities contacted? ☐ Yes ☐ No	Emergency response team activated? ☐ Yes ☐ No		
Cleanup procedures used:			
Cleanup personnel:			
Corrective action required to reduce risk:			
Personnel Signature	Date:		
Manager Signature	Date:		
Chemical Hygiene Officer Signature	Date:		

Additional information on back

Attachment 4 (continued)

Personnel Information

Personnel affected 1.		ID#
2.		ID#
3.		ID#
4,		ID#
Job title 1.	Department: 1.	
2.	2.	
3.	3.	
4.	4.	
Extent of injury		
Describe suspected exposure to hazardous material(s):		
Medical attention required? ☐ Yes ☐ No		
Personnel Signature	Date:	
Manager Signature	Date:	
Chemical Hygiene Officer Signature	Date:	

Attachment 5

COMPUCHEM a division of Liberty Analytical Corp Logbook 18 D 12

Outside 90-Day Storage Area Daily Inspection Log

Date (mm/dd/yy): _____ Inspector: _____

Please check off the following items to verify that each was completed.

☐ Container Condition ☐ Leakage ☐ Bulging ☐ Corrosion ☐ Closed

☐ Lids, caps, & covers on drums

☐ Container properly labeled and dated ☐ Contents indicated on label ☐ "Hazardous Waste" label ☐ Accumulation date on container

☐ Other Container Information ☐ Adequate aisle space present ☐ Contents compatible

☐ Spill kits ☐ Present ☐ Properly labeled

Comments/Corrective Action: _____
_____

Date (mm/dd/yy): _____ Inspector: _____

Please check off the following items to verify that each was completed.

☐ Container Condition ☐ Leakage ☐ Bulging ☐ Corrosion ☐ Closed

☐ Lids, caps, & covers on drums

☐ Container properly labeled and dated ☐ Contents indicated on label ☐ "Hazardous Waste" label ☐ Accumulation date on container

☐ Other Container Information ☐ Adequate aisle space present ☐ Contents compatible

☐ Spill kits ☐ Present ☐ Properly labeled

Comments/Corrective Action: _____
_____

Reviewed by: _____ Date: _____

The presence of the employee's ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible employee together with the employee's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation

Attachment 6

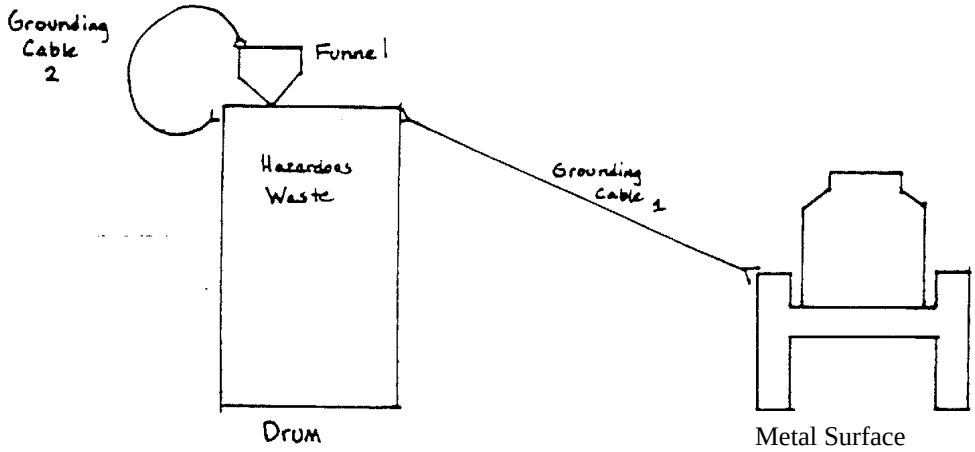
Manifest Tracking Log

CompuChem a Division of Liberty Analytical Corporation

Number	Waste Shipped	Company	Date Sent	Date Received

Reviewed by: _____ Date: _____

Attachment 7

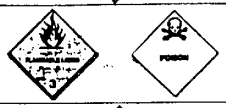
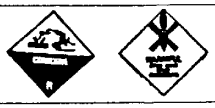


METHOD 1

Fig. 4

Attachment 8

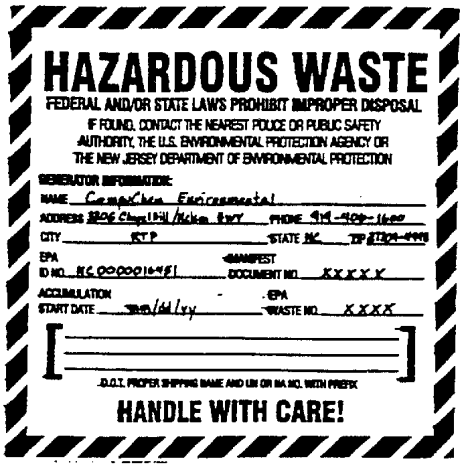
WASTE STREAMS

Profile #	Waste Stream	Code	Shipping Information	RCRA	DOT Label(s)
RVKPU101	Purged Soil Sample	PSSM	Hazardous Waste Solid, NOS (Hexachlorobutadiene, Nitrobenzene) 9, NA 3077, III	F001 F002 F003 F004 F005	
RVKPU102	Flammable Solvent In Vials	SVLS	Waste Flammable Liquid, Poisonous, NOS (Acetone, Methanol) 3, UN 1992, II	D001 F003	
RVKPU103	Plant Scraps	PSWS	Hazardous Waste Solid, NOS (Acetone, Methylene Chloride) 9, NA 3077, III	F002 F003 F004	
RVKPU104	Mixed Flammable Solvents	MFSV	Waste Flammable Liquid, Poisonous, NOS (Methanol, Acetone) 3, UN 1992, II	D001 F003	
RVKPU105	Freon 113	F113	Hazardous Waste Liquid, NOS (1,1,2-Trichlorotrifluoroethane) 9, NA 3082, III	F002	
Left Blank on Purpose					
RVKPU108	019 Water	019W	Hazardous Waste Liquid, NOS (methylene chloride, acetone) 9, NA 3082, III	F002 F003 F005	
RVKPU110	Hardness Reagents	HARD	Ammonium Solution 8, UN 2672, III	D002	
RVKPU111	Ammonia Reagents	AMMO	Waste Corrosive Liquid, NOS (Sodium Hydroxide, Ammonium Chloride) 8, UN 1760, II	D002	
RVKPU112	Phenol Reagents	PHEN	Waste corrosive liquid, poisonous, NOS (Potassium ferrocyanide, boric acid) 8, UN 2922, II	D002	
RVKPU113	Chloride Reagents	CHLR	Waste Corrosive Liquid, Poisonous, NOS (Mercuric thiocyanate, methanol) 8, UN 2922, II	D002	

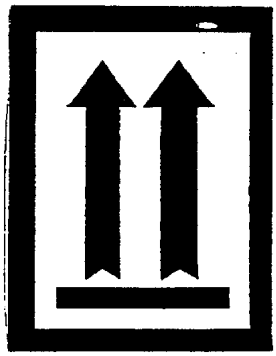
Attachment 9

DOT LABELING SUMMARY

1. Each container of hazardous waste must display a h. label on the top and on one side.



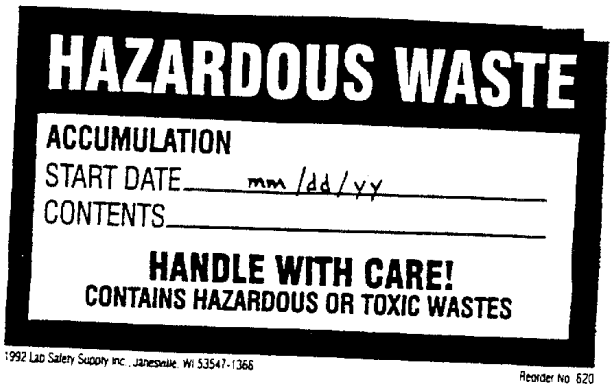
2. Each container of hazardous waste must have two directional labels displayed on opposite sides of the container. When the container is in an upright position, the directional arrows should point upward. Do not affix these labels to the top of the drum!



3. Each container of hazardous waste must have the proper hazard labels ixed. They should be placed on the top of the container and on two sides of container opposite each other. Preferably the labels should be placed next to the labels described above. See page two of this attachment for complete shipping information for each waste stream.

Attachment 9 (continued)

Attachment 9 (continued)



CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ___✓___ outdated procedure (archive) _____

- Procedure Code: ~~Glassware Preparation~~ SOP Section #: 10.2 Revision #: 14

SOP Title: Preparing Glassware for the Inorganics Laboratory

Effective date: (QA fills in) 11/17/05

- Procedure prepared by: Martha Scott Date: 11-17-05
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Pam Stott Date: 11-17-05
- Reason for change: Annual review
- This procedure meets the requirements of the following approved method references: USEPA CLP SOW ILM04.1 and ILM05.3, plus revisions Methods for Chemical Analysis of Water and Wastes, SW-846, Update III, 3rd Edition, 12/96

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

_____ _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Glassware Preparation SOP 10.2: Preparing Glassware for the Inorganics Laboratory

Table of Contents

Glassware Preparation SOP 10.2: Preparing Glassware for the Inorganics Laboratory

1.0 Scope and Application

This procedure describes the proper preparation techniques for glassware used in the inorganics laboratory.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

All glassware is cleaned as soon as possible after use. It is washed in a series of water and/or acid treatments depending on the type of glassware used.

3.0 Definitions

3.1 CLP – Contract Laboratory Program

3.2 SOW – Statement of Work

4.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment and Supplies

5.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20^{th} Edition of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

5.2 Tap water

5.3 1:1 HNO_3 (nitric acid)

5.4 1:1 HCl (hydrochloric acid)

5.5 Detergent-LiquiNox or equivalent

6.0 Sample Collection, Preservation, and Storage

N/A

7.0 Quality Control

7.1 Contamination can occur when glassware, or the material used to clean the glassware, is not thoroughly cleaned. Preventing laboratory contamination is of the utmost concern. Wash Hg digestion glassware separately from glassware used to prep trace metal samples. Keep sinks and drying areas clean. Use fresh hot soapy water for washing each day's glassware, and change drying area paper daily or with each batch of newly washed glassware. Cover clean glassware.

8.0 Calibration and Standardization

N/A

9.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedure.

9.1 **Wash** each beaker with hot tap water and LiquiNox detergent. Volumetric flasks (or any class A glassware) should never be scrubbed. Soak volumetric flask in hot soapy water for approximately half an hour.

9.2 Rinse each piece of glassware thoroughly with hot tap water, then rinse thoroughly with DI water. Place glassware on clean lab bench paper to dry thoroughly, then return the glassware to the Inorganics Preparation laboratory.

9.3 Rinse and swirl all glassware with approximately 10 ml of 1:1 nitric acid (HNO_3) and rinse with DI water.

9.4 Rinse and swirl all glassware with approximately 10 ml of 1:1 hydrochloric acid (**HCl**) and rinse with DI water.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, 3rd edition, Update III, 12/96

11.2 Methods for Chemical Analysis of Water and Wastes, 3/83, Method 200.7

11.3 US EPA CLP SOW **ILM04.1 and** ILM05.3, plus revisions

11.4 New York State Analytical Services Protocol, June 2000

11.5 QCSOP: Proper Documentation Procedures

11.6 QCSOP: Numerical Data Reduction

11.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477

11.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup"

11.9 NELAC Standards, approved **June 2003**, plus revisions

11.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995

11.11 New York State Environmental Laboratory Approval Program, Certification Manual, **April 2005**, plus revisions

11.12 CompuChem Quality Manual, Revision **6**, **Update 1, 5/20/05**, plus revisions

11.13 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

12.0 Attachments as Tables, Diagrams, Flowcharts, and Validation Data

NA

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire shaded area (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure area, title, and SOP number: Section #: 10.1 Effective date: (QA fills in) 4/5/01
  Glassware Preparation SOP: Preparing glassware for the Organic Sample Preparation Lab
- Procedure prepared by T-L Date: 2/12/01
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Linda Carter Date: 3/23/01
- Reason for change: yearly update; NELAC format change
- This procedure meets the requirements of the following approved method references: HAZWRAP, DOE/HWP/65/R1, 7/90; NFESC, 20.2-047B, 6/98; USEPA "Specifications and Guidance for Contaminant-free Sample Containers

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 4/28/02

Annual Review—Signature: _____ Date: 4/3/03

Annual Review—Signature: _____ Date: 1/22/04

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM - ADDENDUM
ANNUAL REVIEW SIGNATURES

This form documents annual review signatures. This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review.

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: 10.1 SOP Section #: _____ Revision #: ____

SOP Title: Glassware Preparation SOP: Preparing Glassware for the Organic Sample Preparation Lab

Effective date: (QA fills in) 4/5/01

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed. The first three annual reviews will appear on the primary SOP Documentation Form. Additional annual reviews are recorded below.

Annual Review—Signature: _____ Date: 2/11/05

Annual Review—Signature: _____ Date: 5/3/06

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

ORIGINAL

Glassware Preparation SOP 10.1: Preparing Glassware for the Organic Sample Preparation Laboratory

Table of Contents

Glassware Preparation SOP 10.1: Preparing Glassware for the Organic Sample Preparation Laboratory

1.0 Scope and Application

This procedure is designed for the preparation of glassware used in the Organic Sample Preparation Laboratory for acid/base/neutral extractables, pesticide/PCB, and herbicide procedures.

2.0 Summary of Method

All glassware is cleaned as soon as possible after use. Concentrator tubes, centrifuge tubes, and other pieces of glassware that hold the final extract should be rinsed with the last solvent used during the extraction. The dirty glassware is brought to the glassware cleaning room and washed in hot, soapy tap water, rinsed with hot tap water, and soaked and rinsed thoroughly with deionized water. The glassware is then drained, put on trays, and placed in the oven where it is annealed for 4 hours at 500° C. Volumetric glassware is not annealed. All continuous liquid-liquid extraction glassware is placed on racks or trays and air dried.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

3.0 Definitions

3.1 anneal – subjecting glass to heat and slow cooling in order to toughen and reduce brittleness.

4.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment & Supplies

5.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in the applicable Instrument Procedure. It is referred to throughout the remainder of this SOP as DI water.

5.2 Chromerge solution – commercially available

5.3 sulfuric acid – concentrated

5.3.1 Prepare the Chromerge sulfuric acid bath by adding one bottle Chromerge to one bottle concentrated sulfuric acid

5.4 nitric acid – 50%, 1:1 v:v with DI water

CAUTION: Gloves, lab coat, safety glasses, and full-face shield must be worn when working with 50% nitric acid or Chromerge solution for cleaning stained glassware.

5.5 Alconox – phosphate-free, biodegradable, commercially available labware cleaning agent

6.0 Sample Collection, Preservation, & Storage

6.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

7.0 Quality Control

7.1 Preventing laboratory contamination is of utmost concern. The following precautions must be taken in order to reduce the possibility of contamination.

7.1.1 Keep all brushes clean and hung on hooks provided.

7.1.2 Change the wash water in the sink when the water becomes dirty. This should occur normally two to three times per shift.

7.1.3 Wash and rinse the sink thoroughly before beginning to prepare the laboratory glassware and before refilling the sink with clean water.

7.2 Glassware inspection

7.2.1 Inspect the glassware for soap or stains that did not come clean during the wash or rinse cycles prior to placing the glassware in the oven for annealing. If this is observed, the cleaning procedure must be repeated. Precleaning treatment may be required.

7.2.2 Check all glassware removed from the oven for stains and breakage. Remove from circulation and discard or repeat the cleaning procedure. Precleaning treatment may be required.

7.3 Pre-cleaning

7.3.1 Before cleaning or if inspection reveals the need, stained glassware may be soaked in 50% nitric acid solution or Chromerge solution to remove stains. After soaking, rinse glassware with tap water, then proceed with normal glassware washing.

CAUTION: Gloves, lab coat, safety glasses, and full-face shield must be worn when working with 50% nitric acid or Chromerge solution for cleaning stained glassware.

7.4 To prevent breakage, the following precautions must be taken.

7.4.1 Do not stack glassware trays directly on top of one another in the oven. This can result in broken glassware.

7.4.2 Do not dump any glassware from wire baskets directly into glassware storage drawers.

7.4.3 Inspect all glassware storage areas for broken pieces of glassware. Remove broken glassware and dispose of properly or send the glassware out to be repaired.

7.4.4 Do not place any glassware on trays in such a manner that the glassware may be broken when the trays are moved in and out of the oven or clean glassware racks.

7.4.5 Broken glassware may be sent out for repair **or discarded in broken glassware receptacles.**

8.0 Calibration & Standardization

NA

9.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedure.

Note: Volumetric glassware (pipets, flasks, etc.) is never annealed.

9.1 Clean all glassware thoroughly as soon as possible after use.

9.2 All sample preparation technicians are required to rinse concentrator tubes, centrifuge tubes, and other pieces of glassware that have held the final solvent extract with the last extraction solvent used.

9.3 Glassware is rinsed with hot tap water prior to immersion in the hot, soapy tap water. Wash glassware with hot, soapy tap water using a phosphate-free, biodegradable detergent (e.g.,**Alconox**).

9.4 Thoroughly brush all glassware. Use proper brushes for each type of glassware and rinse brushes after use.

9.5 Rinse glassware with hot tap water to completely remove all soap residue. Proceed to rinses with DI water.

9.6 Use the DI water **hose** to thoroughly rinse each piece of glassware to ensure that no soap residue remains on the glassware. If any glassware did not come clean during the wash and rinse procedure, the glassware can be soaked in a 50% nitric acid bath for several hours or overnight to remove any staining. Concentrator tubes and other small pieces of glassware can also be placed into the Chromerge sulfuric acid bath to remove stains from the glassware. Any glassware that is soaked in either the 50% nitric acid bath or Chromerge acid bath should be rewashed starting with a tap water rinse.

CAUTION: When adding or removing glassware from either the 50% nitric acid bath or the Chromerge sulfuric acid bath, the glassware preparation technician must wear a full-face shield and two sets of acid-resistant rubber gloves, along with a lab coat and safety glasses.

9.7 Inspect all glassware to ensure that no soap remains on the glassware before placing glassware on trays and putting the trays into the oven. If there are visible signs, continue the rinse process until all traces of soap have been removed.

9.8 **Contaminated glassware should be annealed** in the oven for four hours at 500° C. **Buchner funnels and concentrator tubes should be annealed daily after use.**

9.9 After the oven has cooled, remove the glassware and store it on the appropriate racks to prevent breakage, accumulation of dust, or any other contaminants.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, NaOH, Zn Acetate, or H_2SO_4 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 US EPA "Specifications and Guidance for Contaminant-free Sample Containers."

11.2 US EPA CLP SOW **OLC02.1, OLC03.2, OLM04.2, ILM04.1, ILM05.1, plus revisions**

11.3 "Test Methods for Evaluating Solid Waste; Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96

11.4 QCSOP: Proper Documentation Procedures

11.5 QCSOP: Numerical Data Reduction

11.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

11.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

11.8 NELAC Standards, July 1, 1999, plus revisions

11.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

11.10 New York State Environmental Laboratory Approval Program, Certification Manual, June 2000.

11.11 CompuChem Quality Manual, Revision, **2, 10/26/01** plus revisions

11.12 Sample Control SOP 4.1, "Receiving Samples"

11.13 Sample Control SOP 4.6, "Storing Samples"

12.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

NA

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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: Sample Control SOP Section #: 4.6 Revision #: 19

SOP Title: Storing Samples

Effective date: (QA fills in) 12/02/05

- Procedure prepared by: Valeyna Lepore Date: 11/29/05
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 11/29/05
- Reason for change: Add details of storage units, add contingency protocol
- This procedure meets the requirements of the following approved method references: US EPA CLP SOW OLC03.2, OCM04.3, ILM04.1 and ILM05.3 plus revisions; NYSASP, 6/2000 + revisions; SW-846, 3rd Edition, Update III, 12/96

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Control SOP 4.6: Storing Samples

Table of Contents

Sample Control SOP 4.6: Storing Samples

1.0 Scope and Application

This SOP describes the responsibility of the sample custodian to properly store all samples and check and record the storage cooler temperature daily.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

All received samples and processed extracts are placed in secured cooler storage facility, located in the Sample Control department. Sample storage temperature requirements are different among the agencies that oversee laboratory certifications and approvals. In order to satisfy them all, we choose to exercise the most rigorous, maintaining sample storage temperatures at 2-4.4**^{o}C**. Only aqueous metal samples are allowed to be stored at room temperature in the **receiving area**. (SW-846 extracts are stored at **a** temperature **range** of -10°C to -20°C.**)**

3.0 Definitions

3.1 An SDG is defined by the following, whichever is more frequent:

- **each 20 field samples received within a case, or**
- **each 7 calendar days, beginning with the receipt of the first sample.**

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, and matrix spike duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.**2** CLP – Contract Laboratory Program

3.**3** SOW – Statement of Work

3.4 TCLP – Toxicity Characteristic Leaching Procedure

4.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff is encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment & Supplies

5.1 Sample storage units located in the receiving area

5.1.1 Coolers #3, 5, 6, and 7 store soil samples in the receiving area. These coolers are used for long-term storage of samples after analysis.

5.1.2 Cooler #8 stores aqueous samples in the receiving area. This cooler is used for long-term storage of samples after analysis. Cooler #8 may also be used to store aqueous samples on temporary hold before analysis.

5.1.3 Cooler #5 is used to store aqueous and soil samples in the receiving area that are on temporary hold before analysis.

5.1.4 Cooler #1 (Walk-in) in the receiving area, stores soil and aqueous samples requiring extractable parameters (including TCLP extracts), wet chemistry parameters (includes cyanide digestates), and ILM05.3 metals parameters.

5.1.5 Ambient storage in the receiving area stores aqueous samples for metals parameters (excluding ILM05.3 samples) at room temperature.

5.1.6 Cooler #2A in the receiving area stores wet chemistry samples and GC and GC/MS extracts after analysis.

5.1.7 Freezer #1 in the receiving area stores biological tissue samples and GC and GC/MS extracts on a long-term basis after analysis.

5.2 Sample storage units located in the volatiles laboratory

5.2.1 Refrigerator #4 stores aqueous samples scheduled for OLC03.2 analysis in the volatiles laboratory.

5.2.2 Refrigerator #5 stores soil samples scheduled for analysis in the volatiles laboratory.

5.2.3 Refrigerator #2B stores aqueous samples scheduled for analysis in the volatiles laboratory.

5.2.4 Freezer #6 stores Encore samples and performance evaluation samples in the volatiles laboratory.

5.3 All coolers and refrigerators used for sample storage are maintained at 2^{o}C to 4.4^{o}C. All freezers used for sample and sample extract storage are maintained at –10^{o}C to –20^{o}C.

6.0 Sample Collection, Preservation, & Storage

6.1 Samples collection and preservation requirements are located in Sample Control SOP 4.1: "Receiving Samples."

7.0 Quality Control

7.1 If a temperature reading is unacceptable, follow these steps:

7.1.1 Check the refrigerator fan operation. Call the facilities staff if the fan is not working.

7.1.2 Close the storage **refrigerator** doors and take the temperature reading again in a half hour. If the temperature is not within range, call the facilities manager.

7.1.3 Note who was contacted and what corrective action was taken in the designated columns of the temperature logbook (Attachment 1).

8.0 Calibration & Standardization

8.1 Laboratory thermometers are calibrated annually by the Organic Standards Chemist according to QA SOP 17.12:"Calibrating Thermometers."

8.2 The temperature of each sample cold storage unit is checked daily and recorded in the temperature logbook for that unit. (Attachment 1) The acceptable temperature ranges for the specific cold storage unit is documented in its temperature logbook.

8.2.1 In addition to entering the date, time, temperature and your initials, indicate whether the temperature is acceptable and the whether the thermometer's mercury column is intact.

8.2.2 Any excursions from the acceptable temperature range must be documented along with the corrective actions taken. Corrective action procedures for temperature excursions are documented in the temperature logbooks and posted on the cold storage unit.

8.2.3 Each completed logbook page must be reviewed by the area supervisor or his /her designee.

9.0 Procedure

After samples are received (see SOP 4.2 "Receiving Samples"), they are placed in the appropriate cold or ambient storage unit. Samples are removed from these storage units by authorized personnel for preparation and analysis. Sample transfer is documented on the internal chain-of-custody (COC). (Attachment 2)

9.1 Removing samples from storage for preparation

Samples that undergo a preparation step (extraction, digestion, distillation) are requested from storage using an internal chain-of-custody form.

9.1.1 During first shift hours, the samples are removed from storage by the Sample Custodian and delivered to the preparation lab with a COC documenting samples were relinquished from storage.

9.1.2 The Sample Custodian (time permitting) delivers the samples to the preparation lab with a signed COC at the end of first shift for processing during second shift hours.

9.1.3 The Sample Custodian puts samples requested for processing during weekend hours on a cart in the Walk-in with a COC. The samples remain in the Walk-in until authorized personnel from the preparation lab remove them. Upon removal the COC is signed documenting the day that the samples were relinquished from storage.

9.1.4 If an additional sample is needed from storage during hours that the Sample Custodian is not available, authorized personnel may complete an internal COC form and remove the sample form storage.

9.2 Storing Samples After Preparation and Analysis

9.2.1 After sample preparation, unused sample and empty sample containers are returned to the Sample Custodian. Unused sample is stored in the appropriate storage unit, until it is disposed of (see SOP 4.7 "Organizing and Designating Raw Samples for Disposal").

Note: Sample received for volatile analyses are placed in the refrigerators or freezer in Volatiles lab until analysis is complete. The samples are returned to the Sample Custodian for long-term storage after analysis is complete.

9.2.2 Sample extracts, digestates, or distillates scheduled for analysis by the laboratories, are placed in temporary storage in the appropriate laboratory. After analyses are completed, the extracts, digestates, or distillates are placed into log-term storage by the Sample Custodian, until disposal (see SOP 4.7 "Organizing and Designating Raw Samples for Disposal").

9.3 Contingency

9.31. If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed. For the CLP, the Sample Management Office must be contacted.

9.3.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.3.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.3.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager or for CLP, the Sample Management Office.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3 or H_2SO_4 to pH <2 or NaOH to pH >12 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 US EPA CLP SOW OLC03.2, OLM04.**3**, **SOM01.1,** ILM04.1, **ILM05.3,** plus revisions

11.2 Test Method for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, Update III, 12/96

11.3 New York State Analytical Services Protocol, June 2000**, plus revisions**

11.4 Sample Control SOP 4.1 "Receiving Samples"

11.5 QA SOP 17.12: "Calibrating Thermometers"

11.6 QCSOP: Proper Documentation Procedures

11.7 QCSOP: Numerical Data Reduction

11.8 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

11.9 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

11.10 NELAC Standards, Ju**ne 2003**, plus revisions

11.11 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

11.12 New York State Environmental Laboratory Approval Program, Certification Manual, **April 2005**, plus revisions.

11.13 CompuChem Quality Manual, Revision **7, 11/14/05,** plus revisions

11.14 CompuChem Chemical Hygiene Plan

11.15 Sample Control SOP 4.4 "Sample Custody and Responsibilities of the Sample Custodian"

11.16 Sample Control SOP 4.5 "Ensuring Sample Security"

11.17 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998)

12.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

12.1 Attachment 1 - Cooler Temperature Log Example

12.2 Attachment 2 – Internal Chain of Custody (COCs) Examples

Attachment 1

Sample Receiving Walk-in Refrigerator #1 Temperature Log

CompuChem, a division of Liberty Analytical — Logbook 7 A 4

Acceptable temperature range is 2 °C to 4 °C. Record temperatures to the nearest degree. If temperature is not within acceptance range, notify supervisor or maintenance immediately. Temperature readings should be taken at approximately the same time each day. Please indicate if the time recorded is am or pm.

Daily Temperature Reading						Temperature Corrective Action	Mercury Separation		
Date	Time	Temperature (°C)	Initials	AC	UN	If unacceptable, action taken*	Yes	No	If yes, date rejoined** or new thermometer ID

AC = Acceptable UN = Unacceptable

*Once corrective action is completed, document an additional temperature reading to verify unit is within acceptance criteria.

** If mercury separation in the thermometer is evident, notify supervisor immediately so that the mercury can be rejoined following QA SOP 17.12.

Reviewed by: _____ Date: _____ 10/11/05 jad

Note: Attachment subject to change without notice.

Attachment 2

CompuChem, a Division of Liberty Analytical

INTERNAL CHAIN OF CUSTODY

Laboratory: Volatiles Matrix: W

Request Date: 10/19/2005 Batch 15104

Comments: _____

	CCN	Receipt	Analysis Param	Container	Preservative	Bottle
1	812501	10/19/2005	VW-OLC32X	40MLVIAL	HCL	812501-1
2	812502	10/19/2005	VW-OLC32X	40MLVIAL	HCL	812502-1
3	812503	10/19/2005	VW-OLC32X	40MLVIAL	HCL	812503-1
4	812504	10/19/2005	VW-OLC32X	40MLVIAL	HCL	812504-1
5	812505	10/19/2005	VW-OLC32X	STORAGE	HCL	812505-1

Relinquished By:	Received By:	Date/Time	Reason
#4		10/19 2000	analysis
	#4	10/19 2030	storage

Wednesday, October 19, 2005

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Attachment 2 (Continued)

Wed

Internal Chain of Custody

CompuChem a division of Liberty Analytical Corp. *RAW SAMPLE*

Laboratory: Metals ✓ Requested By: M Hott

Water ✓ Soil _____ P.E. _____ Date: 5-31-05

EPA _____ Commercial _____ Shift: (1) 2 3

This form generated on: 5/31/2005

	CCN	Receipt Date	Preservation	Analysis Parameter	Bottle Number (receiving use only)
1	659901	05/14/05		METALS HG	of
2	659904	5/14/2005		METALS HG	of
3	659905	5/14/2005		METALS HG	of
4	659906	5/14/2005		METALS HG	of
5	659907	5/14/2005		METALS HG	of
6	659908	5/14/2005		METALS HG	of
7	659909	5/14/2005		METALS HG	of
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Relinquished By: _____ Received By: M Hott Date: 6-1-05 Time: 6:55Am

Relinquished By: M Hott Received By: Cooler Date: 6-1-05 Time: 11:40Am

Relinquished By: _____ Received By: _____ Date: _____ Time: _____

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Attachment 2 (Continued)

Internal Chain of Custody

CompuChem a division of Liberty Analytical Corp. *RAW SAMPLE*

Laboratory: Ts Requested By: [signature]

Water ✓ Soil ____ P.E. ✓

Date: 5/19/05

EPA ____ Commercial ✓

Shift: (1) 2 3

This form generated on: 5/19/2005

	CCN	Receipt Date	Preservation	Analysis Parameter	Bottle Number (receiving use only)
1	659901	5/14/2005		METALS HG	of
2	659902	5/14/2005		METALS HG	of
3	659904	5/14/2005		METALS HG	of
4	659905	5/14/2005		METALS HG	of
5	659906	5/14/2005		METALS HG	of
6	659907	5/14/2005		METALS HG	of
7	659908	5/14/2005		METALS HG	of
8	659909	5/14/2005		METALS HG	of
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Relinquished By: [signature] Received By: [signature] Date: 5/19/05 Time: 10 45

Relinquished By: [signature] Received By: [signature] Date: 5/19/05 Time: 11 00

Relinquished By: [signature] Received By: [signature] Date: 5/20/05 Time: 10 00

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Note: Attachment subject to change without notice.

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Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: Sample Control SOP Section #: 4.3 Revision #: 8

SOP Title: Checking & Recording pH of Aqueous Cyanides, Phenols, & Met Chem Samples

Effective date: (QA fills in) 1/23/04

- Procedure prepared by: T. Cannon Date: 1/20/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane Ellsmore Date: 1/23/04
- Reason for change: New COC
- This procedure meets the requirements of the following approved method references: SW846, 3rd Ed., Update III, 12/96; NELAC Standards, 7/99 plus rev.

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: Tammy Cannon Date: 5-26-05

Annual Review—Signature: Cathy Dorey Date: 7-5-06

Annual Review—Signature: _____ Date: _____

Sample Control SOP 4.3: Checking and Recording pH of Aqueous Metals, Cyanides, Phenols, and Wet Chemistry Samples

Table of Contents

Sample Control SOP 4.3: Checking and Recording pH of Aqueous Metals, Cyanides, Phenols, and Wet Chemistry Samples

1.0 Scope and Application

When the Sample Control Clerk receives water samples, he or she must check the pH of those samples preserved with acid or base. This SOP describes the correct procedure for accurately checking and recording sample pH.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

The pH is taken and documented upon sample receipt for a variety of samples to determine that the sample has been adequately preserved. Customer Service contacts the client if the pH is outside the acceptable range, and the client directs the laboratory on how to proceed.

3.0 Definitions

3.1 aliquot - to remove a portion of a sample and put it into a new container.

3.2 QA Notice - Quality Assurance Notice – a record of communication composed by the Quality Assurance Department staff used to notify the client of certain quality issues related to their sample(s).

3.3 pH - A measure of the amount of acidity or alkalinity of a sample based upon a pH number scale.

3.4 pH color guide - a color chart that assigns a specific pH number based upon the color of a pH strip after it has been in contact with a portion of the sample.

3.5 CLP – Contract Laboratory Program

3.6 SOW – Statement of Work

3.7 Sample Control Department - The name may be used interchangeably by the lab with "Receiving Department."

3.8 Wet Chemistry Samples – a generic term used to cover all non-organic preserved tests received. Most tests fitting this description are performed in the wet chemistry department.

4.0 Safety

4.1 Wear the proper protective equipment (eyewear, lab coats, and glasses) when handling samples and checking pH.

4.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment & Supplies

5.1 Paper towels

5.2 pH kit with narrow-range pH paper

5.3 disposable plastic pipets

6.0 Sample Collection, Preservation, & Storage

6.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

7.0 Quality Control

7.1 The pH strip is never dipped into the sample container itself; an aliquot is always poured for taking sample pH measurement.

7.2 If the pH for a sample is within the acceptable range, continue with normal receiving procedures found in Sample Control SOP 4.1.

7.3 If the pH for a sample is not within the acceptable range, place the appropriate QA Notice (Attachment 2) with all other receiving records. Send the receiving records with the QA notice to Project Management. If the client opts to have the sample preserved in house, preservation is documented on the QA notice.

7.4 Sample are then preserved in Receiving, if that option is exercised by the client per the QA Notice. The QA notice is stored with the receiving records for inclusion in the final report.

7.5 For samples requiring pH preservation, check the tables of preservation requirements in Sample Control SOP 4.1, "Receiving Samples."

7.6 If you encounter any other problems, see the Receiving Supervisor or the Senior Receiving Clerk.

8.0 Calibration & Standardization

N/A

9.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedure.

9.1 Place a paper towel on the counter in front of the samples requiring testing.

9.2 Place a small strip of pH narrow range pH paper for the expected range on the paper towel. Use a strip for each sample container to be tested.

9.3 Get a clean, disposable plastic pipet for each sample container to be tested.

9.4 Shake the sample container to thoroughly mix.

9.5 Aliquot a very small amount of sample into the pipet and place a drop or two of sample onto a clean strip of pH paper.

9.6 Compare the color on the pH strip to the pH kit guide.

9.7 Note the sample's pH on the log sheet **and/or the field COC** . (Attachment 1/ **Attachment 2**).

9.8 Dispose of the used pH paper strips, pipets, and paper towels with the normal laboratory trash.

9.10 If the sample pH does not meet the test requirements, complete the QA notice (Attachment 3) and forward it to the Project Manager. The client is contacted to determine the handling of the sample.

9.10.1 If the client wants in-house preservation, the QA notice is returned to Receiving and additional preservative is added and then documented on the QA notice. This notice ultimately returns to the Project Manager and is included in the final report.

9.10.2 If the client wants to resample, the QA notice is marked and the sample is discarded following proper waste handling procedures.

9.11 Contingency

9.11.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.11.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.11.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HNO_3, H_2SO_4, NaOH, or Zn Acetate are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 U.S. EPA CLP SOW OLC03.2, OLM04.3, ILM04.1, ILM05.2 plus revisions

11.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96

11.3 Methods for Chemical Analysis of Water and Wastes, March 1983

11.4 New York State Analytical Services Protocol (NYSASP), June 2000

11.1 QCSOP: Proper Documentation Procedures

11.2 QCSOP: Numerical Data Reduction

11.3 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

11.4 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

11.5 NELAC Standards, **approved May 2001,** plus revisions

11.6 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

11.7 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

11.8 CompuChem Quality Manual, Revision **4, 12/10/03, plus revisions**

12.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

12.1 Attachment 1 - CompuChem Sample Receiving Log

12.2 **Attachment 2 – Field COC**

12.3 Attachment **3** - QA Notice for pH

Attachment 1

COMPUCHEM a Divison of Liberty Analytical Page ______ of ______
COMMERCIAL RECEIVING LOG

Client:
Project:
Quote:
Login No.
Subcontract? Y / N
TAT Verbal Report

Rec'd Date:
Courier:
Airbill No. ____________

PPS/RFA
Lab Instructions

Cooler Rec'd By:
Sample Login By:
Temperature: °C
Cyanide Samples checked for sulfide & chlorine? Y / NA
Phenol Samples checked for chlorine? Y / NA
Received in Good Condition? Y / N
If no, explain:

						Parameters															
CompuChem ID	Client ID	QC	Matrix	Date 20___	Military Time	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH
				/	:																
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Container Type Abbreviations: 40mL(40mL vial) AL(Amber Liter) PL(Plastic Liter) 500P(500mL Plastic) 250P(250mL Plastic) OTHER____________

rl1 - 6/28/01.doc

Attachment 2

CompuChem
a division of Liberty Analytical Corp.

CHAIN OF CUSTODY

501 Madison Ave.
Cary, NC 27513
Phone: 919-379-4100 Fax 919-379-4040

Page ___ of ___

Courier
Airbill No.
Sampling Complete? Y or N

'ompany Name | Project Name
ddress | Sampling Location
ity State Zip | Turnaround time
roject Contact | Batch QC or Project Specific? If Specific, which Sample ID?
hone # | Are aqueous samples field filtered for metals? Y or N
ampler's Name | Are high concentrations expected? Y or N? If yes, which ID(s)?

GW - Ground water
WW - Waste water
SW - Surface water
SO - Soil/Sediment
TB - Trip Blank
RI - Rinsate
WP - Wipe
O - Other

	Field ID	Collection Date	Collection Time	Matrix	# of bottles	Number of Preserved Bottles: HCl	HOPN	HNO3	H2SO4	NaOH	Other

ample Unpacked By:
ample Order Entry By:
amples Received in Good Condition? Y or N
o, explain:

Cyanide samples checked for sulfide & chlorine? Y or NA
625 & Phenol samples checked for chlorine? Y or NA
608 samples checked for pH between 5.0-9.0? Y or NA

elinquished by: Date/Time: Received by: Date/Time:
elinquished by: Date/Time: Received by: Date/Time:
bcontact? Y or N If yes, where? Custody Seal(s) intact? Y or N On Ice? Y or N Cooler Temp: °C

emains stored 60 days after data report mailed at no extra charge.

White & Yellow copy to lab • Pink copy for customer

Attachment 3

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-2
020529

QAN-R-2:052902:llc

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) ~~31~~ 3/21/06

- Procedure Code: Sample Control SOP Section #: 4.1 Revision #: ~~30~~

SOP Title: Receiving Samples — Effective date: (QA fills in) 3/21/06

- Procedure prepared by: Reppas — Date: 3/19/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Alice Evans — Date: 03/21/06
- Reason for change: Annual Review and Attachment Revisions and Additions
- This procedure meets the requirements of the following approved method references: US EPA CLP SOW OLC03.2, OLM04.3, SOM01.1/ILM04.1, and ILM05.3 plus Revisions; SW-846 3rd Edition; Update III 12/96; NYSASP 6/2000 and Revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Control SOP 4.1 Receiving Samples

Table of Contents

Sample Control SOP 4.1 Receiving Samples

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the process of receiving samples from the US EPA and commercial clients. It contains a full description of the log-in procedure.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

After all incoming samples have been received from the carrier at the loading dock, the Sample Control clerk logs them in. When they are received, the sample containers must be checked to ensure that they are intact. Any damage to the sample containers must be noted on the appropriate paperwork. Chains-of-custody (COCs) must be verified against the actual sample received. Sample information is entered into the Laboratory Information Management System (LIMS) for scheduling and tracking purposes. Finally, the samples are placed into cold storage until preparation and analysis.

3.0 Definitions

3.1 Sample Control Department - The name "Receiving Department" or “Shipping and Receiving Department” may be used interchangeably. Staff include the the Supervisor of Sample and Document Control, Project Manager, Sample Control Clerks, and the Assistant EPA Coordinator and/or their assistant.

3.2 LIMS – laboratory information management system.

3.3 NJDEP - New Jersey Department of Environmental Protection

3.4 COC – a chain-of-custody form that arrives with the sample containers. It is located inside the cooler and it identifies the samples, the requested analyses, dates and signatures of people receiving and relinquishing custody of the samples, the number of sample containers, etc.

3.5 Quality Assurance (QA) Notice - used to notify the client and laboratory of any outstanding issues or variances regarding the sample (e.g., sample temperature)

3.6 Horizon Sample Number (HSN) – A laboratory identifier assigned to each sample by the LIMS. The sample is tracked internally by means of the Horizon Sample Number.

3.7 Custodian – designated staff who have responsibility for sample security, storage and custody and who control access to the sample storage units.

3.8 CLP - U.S. EPA Contract Laboratory Program; refers to sample received for analyses as specified in a statement of work.

3.9 NON-CLP - refers to samples received for an analysis other than CLP, commonly referred to in the lab as "commercial."

3.10 Statement of Work - SOW, a document that provides guidelines about sample preparation, analysis, and handling.

3.11 CDP - Client documentation package; contains all of the documentation received with the sample containers in addition to forms completed during sample receipt. A copy of the CDP is stored in Customer Service by case number and the original CDP is stored by receipt date in Sample Control.

3.12 RAS - Routine Analytical Services signifies that the samples are to be processed exactly as presented in a specified SOW (i.e., routinely).

3.13 DAS - Delivery of Analytical Services (formerly SAS) identifies special preparation/analytical instructions not identified in an SOW but requested by the EPA.

3.14 RAS + DAS - signifies that samples are to be processed according to the guidelines specified in an SOW with additional preparation/analytical instructions.

3.15 CSF Gray Envelope (Folder) – A gray (or other colored) metal clasp folder that contains completed receiving documents. Originals are placed into the lowest numbered sample delivery group (SDG). One CSF Gray Envelope is generated for each water and soil organic and inorganic SDG. The CSF Gray Folder applies only to EPA unless requested by commercial clients.

3.16 Customer Service Department – The department includes Customer Service

representatives and/or Project Managers depending on the current staffing requirements.

3.17 NCDENR – North Carolina Department of Environment and Natural Resources

3.18 VOA – volatile organic analysis

3.19 Flexibility Clause – The clause, associated with CLP contracts, allows data users to request minor changes to the current SOW.

4.0 Safety

4.1 Glasses, gloves and lab coats are required to be worn when sample coolers are being unpacked and work is being performed with samples at the hood and when samples are being purged.

4.2 All samples should be treated and handled as if they are hazardous.

4.3 Laboratory staff is encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment and Supplies

5.1 NIST – traceable thermometer with a minimum measurable range of 0-50° C with a calibration tolerance of ± 1.0° C.

5.1.1 Other temperature measurement devices that can be calibrated to ± 1.0° C and have a range of 0-20° C, such as an IR gun.

5.2 narrow range pH paper

5.3 lead acetate test strips

5.4 potassium iodide starch test strips

6.0 Sample Collection, Preservation, & Storage

6.1 Attachment 1 contains the table of sample preservation and holding times.

6.2 See Sample Control 4.6, “Storing Samples” for storage conditions.

7.0 Quality Control/Corrective Action

7.1 Should any problems or questions arise which are not adequately addressed in the text of this SOP, consult your supervisor.

7.2 Discrepancies noted during receipt and log-in are documented in Quality Assurance (QA) notices (Attachments 2-7), on the COC, or in communication logs and discussed with the client before proceeding. Project Manager is notified of the discrepancy, contacts the client for resolution, and provides feedback to the receiving clerk on how to proceed.

7.3 All calibrated thermometers should be labeled with the calibration date, initials of the person performing the calibration, and have a unique identifier, i.e. serial number.

7.3.1 Contact the Organic Standards Chemist if the label is missing from the thermometer.

7.4 Every attempt should be made to complete the sample receiving process so that samples do not remain outside refrigeration for longer than one hour. For samples put on hold or for which there are questions awaiting resolution from the client, put the samples on a cart and wheel them into the walk-in storage unit until further notice.

8.0 Calibration and Standardization

8.1 The Vice President of Quality Assurance and Technology calibrates the IR Gun every three months using the NIST thermometer. Sample Control staff performs a check using three different bottle types (plastic, amber glass, and clear glass). A check of the calibration is performed once a week and is completed by placing a calibrated thermometer into each of the three different pre-chilled bottle types (water filled), and noting the temperature after the thermometer has stabilized. At the same time the thermometer stabilizes, the IR Gun should be placed approximately 13 inches from the bottle being checked and the trigger should be pulled to lock the temperature into the IR Gun's view screen. Each of the temperatures should be noted in Logbook 7 S 1 (IR Gun Check, Attachment 8). The tolerance for the check of the IR Gun is ± 1° C.

8.2 The Organic Standards Chemist calibrates all NIST-traceable thermometers against a NIST thermometer on an annual basis. Records are maintained in the Standards Laboratory. Temperatures of storage units are recorded daily in each of their respective logbooks (Attachment 9).

9.0 Procedures

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The sample coolers are routinely delivered by standard courier such as Federal Express or UPS or may be hand-delivered from the field. All coolers are handled in the following manner.

9.1 Check the cooler and each sample container for damage.

9.1.1 Check the exterior of the cooler for any signs of damage such as a broken seal, signs of sample leakage, etc. Note any observed damage on the commercial COC (Attachment 10) or EPA Organic/Inorganic Traffic Report (Attachments 11 and 12.)

9.1.2 Place the cooler under a hood to open. If this is not possible, place the coolers on the floor next to the hood. When removing the samples place them immediately in the hood.

9.1.2.1 Immediately after opening the cooler, and prior to the removal of any samples, the temperature of the sample shipping cooler is measured as described in Section 9.2 of this SOP.

9.1.2.2 If a sample container is completely broken, therefore unsalvageable, sample volume is properly disposed following Hazardous Waste SOP 12.1, “Hazardous Waste Disposal”.

9.1.2.3 If a sample container is broken or any type of sample spill occurs in the laboratory, clean-up should following guidelines given in Hazardous Waste SOP 12.2, “Spill Control and Cleanup”. .

9.1.2.4 If a soil sample container is broken or cracked and the raw sample has not come into contact with another sample, the sample may be salvageable. The decision of whether to salvage the sample is left to the client. The Project Manager will notify the client of the situation. If the client chooses to continue the analysis, note this information on the COC.

9.2 Checking and recording the temperature of a sample.

9.2.1 Temperature Requirements

9.2.1.1 Commercial clients typically require sample temperature to be 4 ± 2° C. For samples submitted to meet the regulatory requirements

of North Carolina DENR, the temperature tolerance is 2 - 4.4° C. For the US EPA the temperature must not exceed 10° C.

9.2.1.2 The NIST-traceable thermometer or IR gun, must have a minimum measurable range of 0 - 50° C with a calibration tolerance of ± 1.0° C in order to be used.

9.2.1.2.1 Alternate temperature measurement devices may be used if they can be calibrated to ± 1.0° C and have a working range of 0 - 20° C.

9.2.1.3 If a temperature reading is suspicious or does not meet the anticipated temperature range, the temperature should be double checked using an alternate measuring device. For example, if the IR gun returns a temperature below zero but the samples do not appear frozen or feel cold to the touch, then the temperature should be verified using a traceable thermometer or other measuring device.

9.2.1.3.1 If the problem persists, the measuring device should be removed from service until it is re-calibrated.

9.2.1.4 Once a year the Organic Standards Chemist calibrates all laboratory thermometers against the laboratory NIST thermometer. Thermometers are calibrated at their point of use and the NIST thermometer is calibrated at ice point and boiling point. Every three months a calibration of the IR gun is performed.

9.2.2 IR Gun

9.2.2.1 The IR Gun is used for temperature determination. A bottle from the cooler (as close to the center as possible) is used. The temperature must be measured upon opening the cooler and prior to unpacking the samples and removing any packing material.

9.2.2.2 Place the IR Gun approximately 13 inches from the bottle and squeeze the trigger. The bottle temperature will now be locked on the IR Gun's view screen. When aiming the IR Gun towards the bottle, eliminate as much background interference by holding the gun level and making sure that other material surrounding the bottle is not also being checked. Once the temperature is known it should be documented on the COC and log in sheet. All correction factors should be applied.

9.2.2.2.1 If the correction factor for clear glass containers is 1° C and the temperature of a sample in a clear glass container reads 4° C with the IR Gun, then a temperature of 5° C should be recorded on the COC and log in sheet, etc.

9.2.3 Temperature Recording for Coolers received with a Temperature Indicator Bottle (temperature blank)

9.2.3.1 The temperature of the "USEPA Cooler Temperature Indicator," received under the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) is measured with the IR gun. Some non-CLP clients refer to this temperature indicator as a temperature blank.

9.2.3.2 Alternatively, a calibrated NIST-traceable thermometer may be used. Remove the cooler temperature indicator bottle cap, insert the thermometer into the bottle, and allow a minimum of one minute for equilibration before taking the temperature.

9.2.4 Temperature Recording Coolers received without a Temperature Indicator Bottle (temperature blank)

9.2.4.1 If a Cooler Temperature Indicator bottle is not present in an EPA cooler, the EPA Coordinator or Assistant is required to contact the Sample Management Office (SMO), inform them of that fact, and use an alternative means of determining the cooler temperature.

9.2.4.2 The following is a list of options employed by CompuChem to determine the cooler temperature. As required by the CLP SOW documents, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative. Additionally, the QA Notice (attachment 5 and 6) must be included for EPA samples.

9.2.4.3 Options for Water Samples

- An aliquot from a sample bottle designated for extractable organics or metals is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after an approximately one minute equilibration period. The contents of the disposable

container are then properly discarded and not returned to the sample bottle.

- A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

9.2.4.4 Options for Soil Samples

- A calibrated IR gun is used, as indicated for water samples.

9.2.5 Recording Temperature Information

9.2.5.1 Record information on the receiving log (Attachment 13 for EPA organic samples, Attachment 14 for EPA inorganic samples, and Attachment 15 for commercial samples) found in the Sample Control area. Also record the information on the EPA or commercial COC.

9.2.5.2 If the temperature is out of range (4 +/- 2^{o} C for most commercial clients except as stated below), complete the appropriate sections of the required QA Notice (Attachment 2). The Project Manager will notify the commercial client of unacceptable temperature readings so the client can direct CompuChem on how to process the samples.

For samples received out of the range specified by North Carolina (2 - 4.4° C), CompuChem is responsible to notify the State Laboratory of the excursion, if samples have been submitted to meet the regulatory requirements of the NC DENR.

9.2.5.3 For EPA samples, if the Cooler temperature exceeds 10° C, the EPA Coordinator or Assistant contacts SMO and informs them of the temperature deviation. The SMO will obtain directions from the Region that must be documented and included in the SDG Narrative. The narrative information will list, by fraction, the EPA sample number of all samples received in a cooler with a temperature exceeding 10° C. The actual temperature should also be included in the narrative.

The SMO conversation is documented through the use of e-mail correspondence. This process is utilized for all issues or discrepancies.

Also, for EPA CLP dissolved metals samples, review the traffic report or COC for digestion instructions. If there are none, the samples designated as dissolved metals will be digested.

9.3 Verification process for the COC

9.3.1 Remove all paperwork and each sample container and place them on the receiving table under the hood.

9.3.2 Sign your full name and enter the date and time (of delivery by courier) on the COC under "received by laboratory."

9.3.3 Check each sample container for signs of damage. Note any observed damage on the sample's COC.

9.3.4 Verify that all information, including sample identifiers that appear on the sample container, matches the information on the COC, packing list, and traffic sheets, which are located inside the cooler. Note any discrepancies on the COC. Contact the Customer Service representative or Project Manager for non-EPA samples so that they can contact the client for resolution.

9.3.5 If discrepancies are found, the EPA Coordinator or Assistant notifies the Sample Management Office (SMO). The Receiving Clerk must note the discrepancy on the Traffic Report.

9.3.6 On each complete EPA Traffic Report, stamp or write the statement "received in good condition" (or for commercial clients circle the “Y”/”N” option on the receiving log) and initial and date (mm/dd/yy) if the samples meet the following conditions:

- were received intact
- all information on the COC represents actual Cooler contents
- had all associated sample tags
- had all custody seals intact
- had a proper pH for inorganics and wet chemistry samples

- had all corresponding documentation

Note: Sample temperature is not a consideration when using the statement "received in good condition." However, the temperature must be noted on the receiving log and the Traffic Report/COC.

9.3.7 If the samples do not meet the "good condition" criteria established above, make the proper notations on the COC and the receiving log, and initial and date the notations.

9.4 Measuring sample pH

9.4.1 Check the pH of every pre-preserved inorganic, wet chemistry, and organic Method 608 sample, except those requiring analysis for VOA, RSK175 (Methane, Ethane and Ethene), and GRO, following Sample Control SOP 4.3, “Checking and Recording pH of Aqueous Cyanides; Phenols; and Wet Chemistry Samples.”

9.4.2 Record each sample's pH on the receiving log under the pH column, and on the COC. If a sample is not pre-preserved or is a VOA or GRO that cannot be opened due to headspace, record “NA” under the pH column of the receiving log.

9.4.3 If the pH is out of range, complete the appropriate QA notice (Attachment 3). The Project Manager will notify the commercial client of unacceptable pH readings so the client can direct the laboratory on how to process the samples. The EPA Coordinator or Assistant will contact SMO.

9.4.4 For those organic samples to be analyzed by Method 608, which will not be extracted within 72 hours of collection, the pH must be checked and adjusted to between 5.0 and 9.0 at the time of receipt. The pH must be adjusted with NaOH or H_2SO_4 and the amount added must be recorded. The Receiving Clerk makes the pH adjustment.

9.4.5 For samples received which are improperly preserved and have been submitted to meet the regulatory requirements of the NC DENR, the laboratory is responsible for notifying the State Laboratory of the situation. If a replacement sample cannot be obtained, results of the original sample must be qualified.

9.5 Residual chlorine and sulfide checks

9.5.1 Perform a residual chlorine check on all samples that are being analyzed for cyanide and phenol and perform a sulfide check for all samples to be analyzed for cyanide. See Sample Control SOP 4.13, "Handling and Verifying Proper Preservation of Samples Being Analyzed for Cyanide or Phenol". Fill out the appropriate QA Notice (Attachment 4 and Attachment 5) if needed.

9.6 Aqueous volatile check

9.6.1 Check each volatile water sample for air bubbles and headspace (empty space inside the vials; vials must be filled completely with sample). Note any observance of air bubbles or headspace on the COC and the receiving log. Air bubbles larger than the size of a pea should be brought to the attention of the Customer Service representative or Project Manager so that the commercial client can be contacted to obtain instructions on how to proceed. The EPA Coordinator or Assistant will contact SMO.

Note: Pea size bubbles are those not exceeding ¼ inch or 6 mm in diameter. This is defined in SW-846 Chapter 4, Update III, 12/96.

For NJDEP aqueous VOA samples, no air bubbles or headspace is allowed.

9.6.2 In instances where aqueous samples have been submitted to meet the regulatory requirements of NCDENR, the State Laboratory must be notified if there is headspace or large air bubbles. If no replacement sample can be obtained, the results of the original sample must be qualified.

9.7 Completing the process for commercial samples

9.7.1 The Sample Control clerk completes all areas of the Receiving Log and/or commercial COC except for the following areas that are completed by the Customer Service representative for commercial samples.

- ✓ PPS/RFA
- ✓ Lab Instructions
- ✓ Quote
- ✓ Login No.

- ✓ Subcontract
- ✓ TAT
- ✓ Sample Login By
- ✓ CompuChem ID
- ✓ QC

Note: Because the CompuChem COC has all the pertinent information, the receiving log is optional for commercial cooler receipts. However, sometimes a client COC is received and a commercial login sheet is needed.

9.7.2 The Sample Control clerk forwards the paperwork to the Project Manager where the following sample receiving steps are completed.

9.7.2.1 The commercial client is contacted, if necessary, regarding any discrepancies noted upon sample receipt.

9.7.2.2 The Project Manager performs the order entry procedure at which point HSAs are assigned. See Customer Service SOP 5.4, "Order Entry."

9.7.2.3 Project Managers maintain the client documentation package (CDP). A copy of the CDP is scanned as an Adobe Acrobat .pdf file and maintained electronically for reference.

9.7.2.4 The CDP contains "original" paperwork and COCs and are maintained in client/project specific files in Customer Service/Project Management. Client-specific requirements are met for handling of "original" paperwork.

9.7.3 At the request of the commercial client, volatile holding/storage blanks are created as described in Sample Control SOP 4.9, "Preparing and Handling Test Samples." Test samples are routinely generated for EPA samples.

9.7.4 After order entry, bottle labels are printed from the LIMS to the printer in the Receiving department. Receiving clerks label all sample containers with the laboratory-generated labels, leaving as much of the original field label exposed as is possible, particularly the field sample ID. The field sample ID must be carefully checked against the lab-generated label at the

time the labels are placed on the containers.

9.7.4.1 The Project Manager must communicate information pertaining to samples designed for long-term storage.

9.7.4.2 The Project Manager must communicate information pertaining to samples arriving from outside of the continental U.S, and designated quarantine states within the continental U.S. (NC, SC, GA, FL, TN, AR, OK, TX, LA, MS, AL). The project manager communicates this information by entering it into the LIMS. This will then flag the quarantined samples on the purge disposal log.

9.7.5 The labeled samples are placed onto a cart and moved into the secured cooler. Water samples not associated with US EPA CLP SOW ILM05.3 to be analyzed for total and dissolved metals are moved into the secured ambient storage room unless hexavalent chromium is requested.

9.7.5.1 Samples submitted, in order to meet the regulatory requirements of the NCDENR, must be maintained at 2 - 4.4° C, except for aqueous, metals-only samples. For that reason, those samples must be stored in a refrigerator that consistently has been maintained at less than 4.4° C but greater than 2° C.

9.8 Completing the process for EPA samples

Note: Separate receiving logs are required for inorganics and organics and for water and soil matrices.

9.8.1 The Sample Control clerk completes all areas of the receiving log except for the following areas that are completed by the EPA Coordinator or Assistant.

- ✓ Login No./SDG
- ✓ Region
- ✓ Contract No.
- ✓ Case No.
- ✓ TAT

- ✓ Lab Instructions
- ✓ $ Amount
- ✓ Sample No.
- ✓ QC
- ✓ Sample Login by

9.8.1.1 Find the Case/SDG number (located in the EPA receiving log) in the weekly EPA scheduling logbook, and note the order number on the receiving log in the "Login No./SDG" field. If the case number is not located in the logbook, see the EPA Coordinator.

9.8.2 The paperwork is provided to the EPA Coordinator or Assistant who must immediately complete the receiving documentation required for the CSF including the Form DC-1 (Attachment 16). Those steps are detailed in Customer Service SOP 5.8, "Creating the Gray Folder." The EPA Coordinator or Assistant also enters the sample receipt information into the LIMS following Customer Service SOP 5.11, "Order Entry in Horizon." This includes the generation of laboratory bottle labels.

Note: A DC-1 must be completed for each cooler containing EPA samples

9.8.3 Once labels are generated, the Receiving Clerk wraps the appropriate label onto each sample container leaving the field sample ID that appears on the existing label in view, if possible.

9.8.3.1 If the samples are designated for long-term storage, as stated on the COC or by the SMO, place an additional label with the disposal date noted, on each container.

9.8.4 Create volatile holding/storage blanks at the rate of two per SDG and place them in the volatile coolers with the samples. Blanks are created as described in Sample Control SOP 4.9, "Preparing and Handling Test Samples."

9.8.5 Place all labeled samples on a cart and move the samples into the appropriate secured cooler or ambient storage unit.

9.9 Contingency

9.9.1 If, due to a lab accident or to QC failure, a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.9.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.9.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.9.4 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3 or H_2SO_4 to pH <2 or NaOH to pH > 12 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 U.S. EPA CLP SOW OLC03.2; OLM04.3; SOM01.1, plus revisions; ILM04.1; and ILM05.3, plus revisions

11.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96

11.3 Methods for Chemical Analysis of Water and Wastes, March 1983

11.4 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

11.5 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

11.6 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

11.7 QCSOP: Proper Documentation Procedures

11.8 QCSOP: Numerical Data Reduction

11.9 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

11.10 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

11.11 NELAC Standards, approved July 2003, plus revisions

11.12 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

11.13 New York State Environmental Laboratory Approval Program, Certification Manual, April 2005, plus revisions.

11.14 CompuChem Quality Manual, Revision **7**, Update 1, **12**/**13**/05, plus revisions

11.15 Sample Control SOP 4.6, "Storing Samples"

11.16 Sample Control SOP 4.3, "Checking and Recording pH of Aqueous Cyanides, Phenols, and Wet Chemistry Samples

11.17 Sample Control SOP 4.9, "Preparing and Handling Test"

11.18 Sample Control SOP 4.13, "Handling and Verifying Proper Preservation of Samples Being Analyzed for Cyanide or Phenol"

11.19 Customer Service SOP 5.8, "Creating the CSF Gray Folder."

11.20 Customer Service SOP 5.11, "Order Entry in Horizon"

12.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

Attachment 1 – Tables of Preservation and Holding Times

Attachment 2 – Quality Assurance Notices for Temperature Excursion

Attachment 3 – Quality Assurance Notice for pH

Attachment 4 – Quality Assurance Notice for Cyanide Samples

Attachment 5 – Quality Assurance Notice for Phenol

Attachment 6 – Quality Assurance Notice for Cooler Temperature Options – Organics

Attachment 7 – Quality Assurance Notice for Cooler Temperature Options – Inorganics

Attachment 8 – Logbook 7 S 1, IR Gun Check

Attachment 9 – Temperature Log Book Example

Attachment 10 – Chain-of-Custody Record

Attachment 11 – EPA Organic Traffic Report Example

Attachment 12 – EPA Inorganic Traffic Report Example

Attachment 13 – EPA Receiving Log - organic

Attachment 14 – EPA Receiving Log - inorganic

Attachment 15 – Commercial Receiving Log

Attachment 16 – Sample Receiving Log (Form DC-1), Organics & Inorganics

Attachment 17 – QA Notices for North Carolina Only

Attachment 1

Table 4.1 Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the Clean Water Act, 40 CFR 136, *Federal Register*

Parameter	Preservation	Holding[a] Time (days)	Containers[b]	Volume (ml)
Acidity	Cool, 4°C	14	P or G	200
Alkalinity	Cool, 4°C	14	P or G	100
Ammonia	Cool, 4°C	28	P or G	500
Biochemical Oxygen Demand	Cool, 4°C	48 hours	P or G	1000
Bromide	NR[c]	28	P or G	200
Chemical Oxygen Demand	Cool, 4°C, H_2SO_4 to pH<2	28	P or G	100
Chloride	NR	28	P or G	100
Chlorine, Total Residual	NR	0[d]	P or G	500
Chromium VI	Cool, 4°C	24 hours	P or G	500
Coliform, fecal and total	Cool, 4°C; 0.008% $Na_2S_2O_3$	6 hours	P or G	200
Color	Cool, 4°C	48 hours	P or G	500
Cyanide, total	Cool, 4°C; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	1000
Cyanide, amenable to chlorination (free)	Cool, 4°C; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	500
Fluoride	NR	28	P	500
Hardness	HNO_3 to pH<2 H_2SO_4 to pH <2	180	P or G	250
Hydrogen Ion (pH)	NR[c]	0[d]	P or G	40
Kjeldahl Nitrogen	Cool, 4°C; H_2SO_4 to pH <2	28	P or G	1000
Mercury	HNO_3 to pH<2	28	P or G	500
Metals (except Cr VI and Hg)	HNO_3 to pH<2	180	P or G	500
Nitrate (as N)	Cool, 4°C	48 hours	P or G	100
Nitrite (as N)	Cool, 4°C	48 hours	P or G	50
Nitrate-Nitrite (as N)	Cool, 4°C; H_2SO_4 to pH <2	28	P or G	500
Oil and Grease	Cool, 4°C; H_2SO_4 to pH <2	28	G	1000
Organic Carbon, Total (TOC)	Cool, 4°C HCl or H_2SO_4 to pH <2	28	P or G	100
Organic Nitrogen, Total	Cool, 4°C; H_2SO_4 to pH <2	28	P or G	500
Orthosphosphate (as P)	Filter immediately, Cool, 4°C	48 hours	P or G	50
Phenols (Total)	Cool, 4°C; H_2SO_4 to pH <2	28	G	1000
Phosphorus (Elemental)	Cool, 4°C	48 hours	G	500
Phosphorus (Total)	Cool, 4°C H_2SO_4 to pH <2	28	P or G	200
TRPH	5 ml HCl; Cool, 4°C	48 hours	G	1000

[a] From time of sample collection
[b] Polyethylene (P) or glass (G)
[c] None required
[d] 0 days indicates that the sample must be analyzed immediately.
[e] Reduced to 24 hours if sulfide is present, unless sulfide is removed before preservation.

Attachment 1 (Continued)

Table 4.1 (Continued) **Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the Clean Water Act, 40 CFR 136, *Federal Register***

Parameter	Preservation	Holding Time[a] (days)	Containers[b]	Volume (ml)
Solids (Total and Filterable)	Cool, 4°C	7	P or G	100
Solids (Non-filterable)	Cool, 4°C	7	P or G	200
Solids (Settleable)	Cool, 4°C	48 hours	P or G	1000
Silica	Cool, 4°C	28	P	100
Specific Conductance	Cool, 4°C	28	P or G	250
Sulfate	Cool, 4°C	28	P or G	250
Sulfide	Cool, 4°C Add zinc acetate and NaOH to pH>9.	7	P or G	
Sulfite	NR[c]	0[d]	P or G	250
Surfactants	Cool, 4°C	48 hours	P or G	250
Turbidity	Cool, 4°C	48 hours	P or G	250
Purgeable Halocarbons	Cool, 4°C 0.008% $Na_2S_2O_3$[e]	14	G	80
Purgeable Aromatic Hydrocarbons	Cool, 4°C 0.008% $Na_2S_2O_3$[f] HCl to pH<2	14	G	80
Phenols	Cool, 4°C 0.008% $Na_2S_2O_3$[f]	7/40[g]	P or G	2000
Benzidines	Cool, 4°C 0.008% $Na_2S_2O_3$[f]	7/7[h]	P or G	2000
Phthalate Esters	Cool, 4°C	7/40[g]	G	2000
Nitrosamines	Cool, 4°C, dark 0.008% $Na_2S_2O_3$[f]	7/40[g]	P or G	2000
PCBs	Cool, 4°C	7/40[g]	P or G	2000
Nitroaromatics and isophorone	Cool, 4°C, dark 0.008% $Na_2S_2O_3$[f]	7/40[g]	P or G	2000
Polynuclear Aromatic Hydrocarbons	Cool, 4°C, dark 0.008% $Na_2S_2O_3$[f]	7/40[g]	G	2000
Haloethers	Cool, 4°C 0.008% $Na_2S_2O_3$[f]	7/40[g]	P or G	2000
Chlorinated Hydrocarbons	Cool, 4°C	7/40[g]	P or G	2000
TCDD	Cool, 4°C 0.008% $Na_2S_2O_3$[f]	7/40[g]	P or G	2000
Pesticides	Cool, 4°C; pH 5-9	7/40[g]	P or G	2000

[a] From time of sample collection
[b] Polyethylene (P) or glass (G)
[c] None required
[d] 0 days indicates that the sample must be analyzed immediately.
[e] $Na_2S_2O_3$ added if residual chlorine is present; approx. equal to 1 ml of 10% thiosulfate / liter of sample.
[g] To complete extraction (or initiate extraction if continuous liquid-liquid extraction is performed)/ to complete analysis following extraction
h Extracts may be stored up to 7 days before analysis if stored under an inert (oxidant-free) atmosphere.

Attachment 1 (Continued)

Table 4.2 **Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Inorganics Analysis, ILM04.0-.1**

Parameter	Preservation[a]	Holding Time[b] (days)	Containers[c]	Volume (ml)
Cyanide, total	Cool, 4°C NaOH to pH>12 0.6 g of ascorbic acid[d]	12	P or G	1000
Metals (except Hg)	HNO_3 to pH<2	180	P or G	500[e]
Mercury	HNO_3 to pH<2	26	P or G	500[e]

a Water sample only; preservation performed by sampler immediately upon sample collection. Soil/sediment samples are maintained at 4°C until analysis. Dissolved metals are filtered onsite by sampler before addition of preservative.
b From validated time of sample receipt
c Polyethylene (P) or glass (G)
d Only used in the presence of residual chlorine
e Can be combined into a one-liter bottle.

Table 4.3 **Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Organics Analysis OLM03.2 (aqueous only), OLM04.2, and OLC02.1 (aqueous only)**

Parameter	Preservation	Holding Time (days)[a]	Containers[b]	Volume (ml)
Aqueous Volatiles	Cool, 4°± 2°C[c] HCl to pH≤2	10[d]	G-TLSSL	80
Soil/Sediment Volatiles	4°± 2°C[c]	10[d]	G-TLC, CET, **EnCore™ samplers**	4 oz
Aqueous Semivolatiles	4°± 2°C[c]	5/40[e]	G	2000
Soil/Sediment Semivolatiles	4°± 2°C[c]	10/40[e]	G	8 oz
Soil/Sediment Pesticides/PCBs	4°± 2°C[c]	10/40[e]	G	8 oz
Aqueous Pesticides/PCBs	4°± 2°C[c]	5/40[e]	G	2000

a From validated time of sample receipt
b All containers are 1-liter glass bottles with Teflon-lined cap except aqueous volatiles (G-TLSSL = 40-ml glass bottle with Teflon-lined septum sealed lid), and soil/sediment volatiles (G-TLC = 4-oz glass jar with Teflon-lined cap or CET = closed-end tubes such as brass sleeves). Soil samples may also be collected under OLM04.2 for low level analysis as 5 gm sample in sodium bisulfate or medium level analysis in pre-weighed vials containing 10ml methanol. When this occurs, the sample vial, with 10 ml methanol and all labeling, is weighed to the nearest 0.1g prior to the addition of sample. Approximately 5g of sample is added to the vial. The sample vial with
sample is weighed to the nearest 0.1g. The initial weight, final weight and sample weight are recorded and provided to the laboratory.
c Preserve samples at time of collection; samples should be stored in the dark until extraction/analysis.
d Until analysis
e To complete extraction/to complete analysis following extraction

Attachment 1 (Continued)

Table 4.4 **Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Aqueous and Solid Matrices[a] Published in SW846 Test Methods for Evaluating Solid Waste, Third Edition, Update 3, December 1996.**

Parameter	Methods	References	Container	Preservation	Maximum Holding Times[b]
Volatile Organics in water	Purge and trap GC and GC/MS	8021B, 8015B, 8260B	Glass, 40-ml vial with zero headspace	4°C, HCl to pH <2, 0.008% $Na_2S_2O_3$	14 days (A) (see below for soil)
Semivolatile Organics	GC, HPLC, and GC/MS	8081A, 8141A, 8151A, 8310, 8082, 8270C	Glass, amber (A) (1 liter sample); 8-oz wide mouth with PTFE-lined cap (S) (50-g sample)	Cool to 4°C, 0.008% $Na_2S_2O_3$	7 days to extract (A); 14 days to extract (S); 40 days to analyze (A/S)
Total Metals – except Mercury and Chromium VI	Furnace AA and ICP	7000 series/ 6010B	Glass or plastic 500ml (A), 8-oz wide mouth (S) (200-g sample)	HNO_3 to pH<2 (A); filter dissolved on-site first; filter suspended-no acid	6 months (A/S)
Chromium VI	Colorimetric, automated	7196A	Glass or plastic, 500ml (A), 8-oz wide mouth (S) (200-g sample)	Cool to, 4°C	24 hours (A); one mo. to extraction, 4 days after (S).
Mercury	Manual cold vapor AA	7470/7471A	Glass or plastic, 500ml (A), 8-oz wide mouth (200-g sample)	pH<2 HNO_3 (A); filter dissolved; Cool to 4°C (S)	28 days (A/S)
Chloride	Colorimetric, automated	9251	Glass or plastic	None required	28 days (A)
Cyanide	Colorimetric automated	9012A	Glass or plastic	Cool to 4°C; pH >12 NaOH (A)	14days (A)
Phenols	Colorimetric, automated	9066	Glass or plastic	Cool to 4°C; pH <4 H_2SO_4 (A)	28 days (A)
Specific conductance	conductivity	9050A	Glass or plastic	Cool to 4°C (A)	28 days (A)
Sulfate	turbidimetric	9038	Glass or plastic	Cool to 4°C (A)	28 days (A)
Sulfide	distillation, titration	9034	Glass or plastic	Cool to 4°C; Zinc acetate (A/S)	7 days (A/S)
Oil & grease	Gravimetric	9070/9071A	Glass	Cool to 4°C (A/S); 5 ml diluted HCl(A)	28 days (A) 14 days (S)
TOC	Combustion analyzer	9060	Glass or plastic	Cool to 4°C; pH<2 HCl or H_2SO_4; Store in dark (A)	28 days
TOX	DX 208 analyzer	9020B	Glass, PTFE-lined cap	Cool to 4°C; pH<2 H_2SO_4	

[a] Table originally excerpted, in part, from Table II, 49 FR 28, October 26, 1984, and revised.
b Holding time begins at time of sample collection.
(A) aqueous
(S) solid

Attachment 1 (Continued)

Table 4.5 Sample Handling, Preservation and Holding Time Table for SW846 Method 5035,Volatile Organics in Soil

Conc. Level	Sampling Device	Collection Procedure	Container Type	Container Preparation	Preservation	Maximum Holding Time[a]
≤ 200 ug/kg	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	4°C, $NaHSO_4$	14 days
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1[b]	4°C	48 hours
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	4°C/-10°C[d]	48 hours/ 14 days[e]
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[b,f,g]	4°C	48 hours
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[f,g]	$NaHSO_4$, 4°C	48 hours, 14 days
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[b,f,g]	4°C/-10°C[c,d]	48 hours/ 14 days
> 200 ug/kg[h]	Encore™ or equivalent	5035-Section 6.2.2.3[f]	Encore™ or equivalent	5035-6.1.1[f,g]	4°C	48 hours 14 days
	Coring Device	5035-Section 6.2.2.3[I]	Glass vial w/ PTFE-silicon septum	5035-6.1.1[I]	Methanol/PEG 4°C	14 days
	Conventional Devices	FLDEP SOP –Section 4.3	Glass vial w/ PTFE-silicon septum	5035-6.1.1	4°C	14 days
Dry weight	Conventional Devices		Glass w/ teflon liner		4°C	

a Maximum time allowed from time/date of collection to sample analysis.
b Eliminate 6.1.1.2; use only organic-free water.
c Contents of sampling device must be transported to the laboratory at 4°C and stored at –10°C; this option upon client request.
d In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle.
e Maximum allowed time at 4°C is 48 hours; maximum allowed time to sample analysis is 14 days from collection.
f Conducted in the laboratory.
g Entire contents of sampling device is extruded into the sample analysis vial containing the appropriate solvent.
h Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200ug/kg.
I Methanolic preservation in the field is not recommended.

Attachment 1 (Continued)

Table 4.6 **Requirements for Holding Times for Hazardous Characteristics Indicators Published in SW846 Test Methods for Evaluating Solid Waste, Third Edition, Update 3, December 1996.**

Parameter	Methods	References	Container	Preservation	Maximum Holding Times[c]
Reactivity-Total releasable cyanide	Reflux distillation, colorimetry	Chapter 7, 7.3.3, 9012A	Glass, with zero headspace	4°C in dark, pH 12 NaOH	ASAP
Reactivity-Total releasable Sulfide	Acid distillation, titration	Chapter 7, 7.3.4, 9034	Glass, with zero headspace	4°C in dark, pH 12 NaOH; zinc acetate	ASAP
Ignitability	Flash point	1010	Glass or plastic	NA	ASAP
Corrosivity	Electrometric	9040B	Glass or plastic	NA	ASAP
Paint filter liquids	filtration	9095A	Glass or plastic	NA	ASAP
EP Toxicity	Leachate generation	1310	Glass or plastic	NA	ASAP

Table 4.7 **Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Toxicity Characteristics Leaching Procedure, TCLP, Method 1311, and Synthetic Precipitation Leaching Procedure, SPLP, Method 1312, Published in SW846 Test Methods for Evaluating Solid Waste, Third Edition, Update 3, December 1996.**

Sample Maximum Holding Times

Parameter	From: Field to collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative Analysis	Total elapsed time in days
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, no Hg	180	NA	180	360

Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

Holding time associated with the ASTM leachate generation method (D3987-85) is 14 days from collection to leachate generation, i.e. filtration, then the method holding time to preparation and/or analysis.

Attachment 2

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

Client _____
Case # _____
Type of Analysis _____
Receipt Date _____

For some organic and/or inorganic determinations temperature preservation at 4 degrees Celsius is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler.

The EPA CLP program requires the laboratory make notification when the temperature exceeds 10^{o} C.

The State of North Carolina requires that samples must be iced to above freezing but $\leq 6^{o}$ C during shipment.

Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____ **Date** _____

QAN-R-3
060308

qanr3 – 3/8/06:jad

Attachment 3

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-2
020529

QAN-R-2:052902:llc

Attachment 4

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-1
971022

Qanr1 – 10/22/97:llc

Attachment 5

CompuChem
a division of Liberty Analytical Corporation

Quality Assurance Notice

CompuChem ID#_____
Client ID#_____
Case #_____
SDG# _____
Receipt Date_____
Method_____

A chlorine check was performed on the above phenol sample and was determined to be present. A member of CompuChem's Customer Service Department contacted the client. The Receiving Department was instructed as follows:

Analyze – Qualify with notice and address in narrative: _____
Dispose – Client will resample: _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-4
010702

Qanr4 – 07/2/01:dce

Attachment 6

CompuChem
a division of Liberty Analytical Corporation

Quality Assurance Notice

Case # _____SDG# _____
Receipt Date _____Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration (Document Number OLM04.3, OLC03.2 and SOM01.1),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples

___An aliquot from a sample bottle designated for extractable organics is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3-minute equilibration period. The contents of the disposable container are then properly discarded.

___A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples

___A calibrated IR gun is used, as indicated for water samples.
___A temperature strip is affixed to the outside of a sample container and, after one minute, the temperature is read and recorded.

As required by the organic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-5
011324

Qanr5 – 11/15/05:vr

Attachment 7

CompuChem
a division of Liberty Analytical Corporation

Quality Assurance Notice

Case # _____SDG# _____
Receipt Date _____Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (Document Number ILM04.1 and ILM05.**3**),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples

___An aliquot from a sample bottle is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3 minute equilibration period. The contents of the disposable container are then properly discarded.

___A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples

___A calibrated IR gun is used, as indicated for water samples.
___A temperature strip is affixed to the outside of a sample container and, after one minute, the temperature is read and recorded.

As required by the inorganic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-6
051115

Qanr6 – **11/15/05**:**vr**

Attachment 8

CompuChem a division of Liberty Analytical Corp. Logbook 7 S 5

IR Gun Check

The tolerance for the IR Gun check is ± 1°C. If this criteria is not met, the IR Gun must be removed for recalibration.
IR calibration performed once per week.

Date	IR Gun No.	Thermometer Temp. °C	Container Type	Container Temp °C	Checked By	Comments
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			

Reviewed By: ________________________ Date: ____________

3/21/03:dce

Attachment 9

Sample Receiving Walk-in Refrigerator #1 Temperature Log

CompuChem, a division of Liberty Analytical Logbook 7 A 4

Acceptable temperature range is 2 °C to 4 °C. Record temperatures to the nearest degree. If temperature is not within acceptance range, notify supervisor or maintenance immediately. Temperature readings should be taken at approximately the same time each day. Please indicate if the time recorded is am or pm.

Daily Temperature Reading						Temperature Corrective Action	Mercury Separation		
Date	Time	Temperature (°C)	Initials	AC	UN	If unacceptable, action taken*	Yes	No	If yes, date rejoined** or new thermometer ID

AC = Acceptable UN = Unacceptable

*Once corrective action is completed, document an additional temperature reading to verify unit is within acceptance criteria.

** If mercury separation in the thermometer is evident, notify supervisor immediately so that the mercury can be rejoined following QA SOP 17.12.

Reviewed by: _____ Date: _____ 10/11/05:jad

Attachment 10

CompuChem
a division of Liberty Analytical Corp.

CHAIN OF CUSTODY

501 Madison Ave.
Cary, NC 27513
Phone: 919-379-4100 Fax 919-379-4040

Page ____ of ____

Courier
Airbill No.
Sampling Complete? Y or N

Company Name	Project Name
Address	Sampling Location
City State Zip	Turnaround time
Project Contact	Batch QC or Project Specific? If Specific, which Sample ID?
Phone #	Are aqueous samples field filtered for metals? Y or N
Sampler's Name	Are high concentrations expected? Y or N? If yes, which ID(s)?

GW - Ground water
WW - Waste water
SW - Surface water
SO - Soil/Sediment
TB - Trip Blank
RI - Rinsate
WP - Wipe
O - Other

	Field ID	Collection Date	Collection Time	Matrix	# of bottles	Number of Preserved Bottles HCl	NaOH	NHO3	H2SO4	MEOH	Other

Sample Unpacked By:
Sample Order Entry By:
Samples Received in Good Condition? Y or N
If no, explain:

Cyanide samples checked for sulfide & chlorine? Y or NA
625 & Phenol samples checked for chlorine? Y or NA
608 samples checked for pH between 5.0-9.0? Y or NA

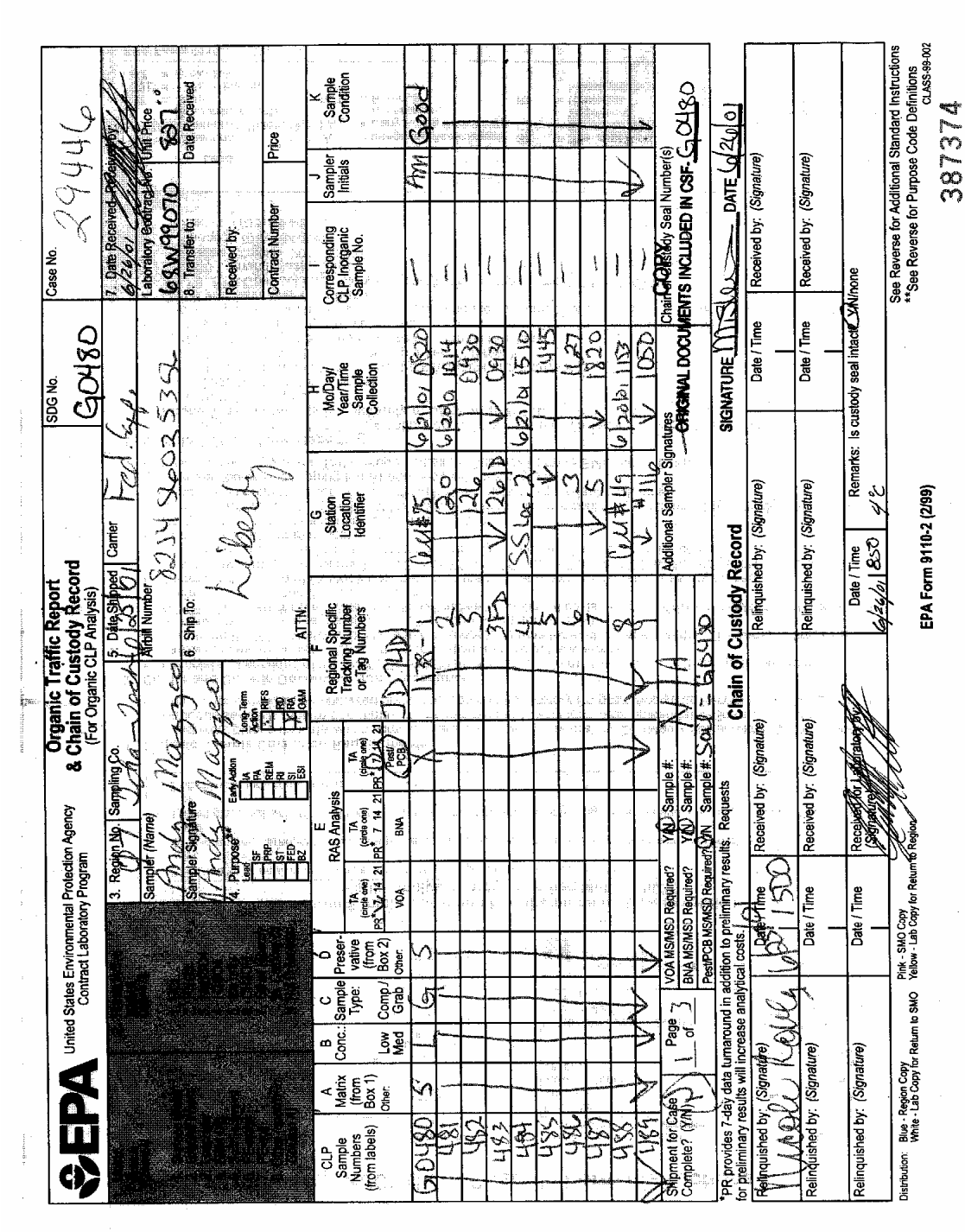
Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:

Subcontact? Y or N If yes, where? | Custody Seal(s) intact? Y or N | On Ice? Y or N | Cooler Temp: °C

Samples stored 60 days after date report mailed at no extra charge.

White & Yellow copy to lab • Pink copy for customer

Attachment 11



EPA — United States Environmental Protection Agency
Contract Laboratory Program

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

SDG No. GD480
Case No. 29446

3. Region No. 07
Sampling Co. SAIC-Jacksonville
5. Date Shipped 6/28/01
Carrier Fed. Exp.
7. Date Received 6/29/01
Laboratory Accession No. 69W99070
Unit Price 897.00

Sampler (Name) Andy Mazzeo
Airbill Number 8234 3e03 5352
8. Transfer to:
Date Received

Sampler Signature Andy Mazzeo
6. Ship To: Lieberty
Received by:

4. Purpose: Early Action (SF, PRP, ST, FED, BZ); Long-Term Action (IA, FA, REM, RI, SI, ESI, PRPS, RD, RA, O&M)
ATTN:
Contract Number
Price

CLP Sample Numbers (from labels)	A Matrix (from Box 1)	B Conc.: Low Med	C Sample Type: Comp/ Grab	D Preservative (from Box 2)	E RAS Analysis: TA (circle one) PR 7 14 21 VOA	E RAS Analysis: TA (circle one) PR 7 14 21 BNA	E RAS Analysis: TA (circle one) PR 7 14 21 Pest/PCB	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Corresponding CLP Inorganic Sample No.	J Sampler Initials	K Sample Condition
GD480	S	L	G	S	X		X	1138-1	104B5	6/26/01 0820	—	AM	Good
481								2	30	6/26/01 1014	—		
482								3	126	1 0930	—		
483								3FB	126D	0930	—		
484								4	SSLoc.2	6/27/01 1510	—		
485								5	1	1445	—		
486								6	3	1427	—		
487								7	S	1820	—		
488								8	104#49	6/28/01 1138	—		
489	V	V	V	V	V	V	V	V	111e	V 1050	—	V	V

Shipment for Case Complete? (Y/N) Y — Page 1 of 3
Chain of Custody Seal Number(s)
Additional Sampler Signatures
ORIGINAL DOCUMENTS INCLUDED IN CSF. COPY

VOA MS/MSD Required? Y/N Sample #:
BNA MS/MSD Required? Y/N Sample #:
Pest/PCB MS/MSD Required? Y/N Sample #: Soil = 5654D

*PR provides 7-day data turnaround in addition to preliminary results. Requests for preliminary results will increase analytical costs.

Chain of Custody Record

SIGNATURE [illegible] DATE 6/29/01

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[illegible]	6/29/01 1500				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	
			6/29/01 1850	4°C	

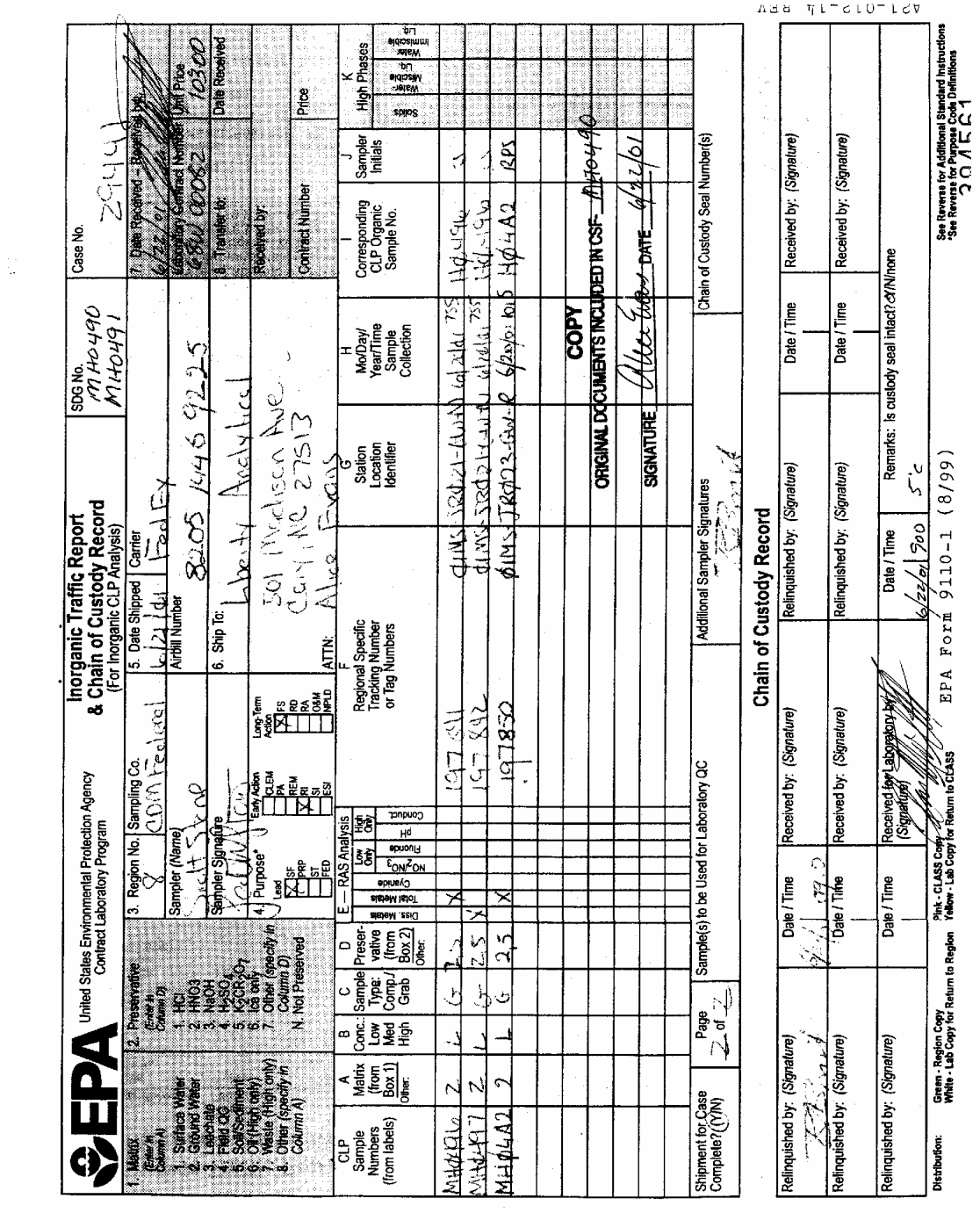
Distribution: Blue - Region Copy; White - Lab Copy for Return to SMO; Pink - SMO Copy; Yellow - Lab Copy for Return to Region

EPA Form 9110-2 (2/99)

See Reverse for Additional Standard Instructions
**See Reverse for Purpose Code Definitions
CLASS-99-002

387374

Attachment 12



Attachment 13

Page ___of ___

CompuChem, a Division of Liberty Analytical Corp.- EPA ORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
Samples Logged in by:	SDG #:
Number of TR's?	Work Order No.:
Return Airbill in cooler? Yes / No	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	T. VOC	VOC	VOC SIM	SVOC	SIM	PEST	AR.	Amount/Containers
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														
13														
14														
15														
16														
17														
18														
19														
20														

OrgRec.log.doc-02/24/06

Attachment 14

Page ___of

CompuChem, a Division of Liberty Analytical Corp.- EPA INORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp. Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
SAMPLE LOGIN:	SDG #:
Number of TR's?	Work Order No:
Return Airbill in Cooler? Yes / No	
Cyanide Samples checked for sulfide & chlorine? YES / NA	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	Total Metals	Cyanide	Dissolved Metals	pH	Amount/Containers
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

InorgRaclog.doc-03/01/06

Attachment 15

COMPUCHEM a Divison of Liberty Analytical **Page _____ of _____**

COMMERCIAL RECEIVING LOG

Client:	Rec'd Date:	PPS/RFA
Project:	Courier:	Lab Instructions
Quote:	Airbill No. ______________	____________________________
Login No.	____________________	____________________________
Subcontract? Y / N	____________________	____________________________
TAT Verbal ________ Report		____________________________

Cooler Rec'd By:
Sample Login By:
Temperature: °C
Cyanide Samples checked for sulfide & chlorine? Y / NA
Phenol Samples checked for chlorine? Y / NA
Received in Good Condition? Y / N
If no, explain:

						Parameters															
CompuChem ID	Client ID	QC	Matrix	Date 20____	Military Time	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH	No. & Type	pH
				/	:																
				/	:																
				/	:																
				/	:																
				/	:																
				/	:																
				/	:																
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				/	:																
				/	:																

Container Type Abbreviations: 40mL(40mL vial) AL(Amber Liter) PL(Plastic Liter) 500P(500mL Plastic) 250P(250mL Plastic) OTHER______________________ rl1 - 6/28/01:dce

Attachment 16

SAMPLE LOG-IN SHEET

Lab Name:					**Page_____of_____**
Received By(Print Name)					**Log-in Date**
Received By(Signature)					
Case Number	**Sample Delivery Group No.**			**Client Number NA**	
Remarks:		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1.Custody Seal(s)	Present/Absent*	1			
	Intact/Broken	2			
2.Custody Seal Nos.		3			
		4			
3.Chain-of-Custody Records	Present/Absent*	5			
		6			
4.Traffic Reports or Packing Lists	Present/Absent*	7			
		8			
5.Airbill	Airbill/Sticker	9			
	Present/Absent*	10			
6.Airbill No.		11			
		12			
7.Sample Tags	Present/Absent*	13			
Sample Tag Numbers	Listed/ Not Listed on	14			
	Chain-of-Custody	15			
8.Sample Condition	Intact/Broken*/Leaking	16			
		17			
9.Cooler Temperature	Present/Absent*	18			
Indicator Bottle		19			
10.Cooler Temperature		20			
		21			
11.Does information on custody	Yes/No*	22			
records, traffic reports, and		23			
sample tags agree?		24			
12.Date Received at Lab		25			
		26			
13.Time Received		27			
Sample Transfer		28			
Fraction	Fraction	29			
Area#	Area#	30			
By	By	31			
On	On	32			
*Contact SMO and attach record of resolution.		33			
Reviewed BY		**Logbook No.**			
Date		**Logbook Page No.**			

Form DC-1 OLC03.2

Attachment 16 (Continued)

SAMPLE LOG-IN SHEET

Lab Name:				**Page_____of_____**	
Received By(Print Name)				**Log-in Date**	
Received By(Signature)					
Case Number	**Sample Delivery Group No.**			**SAS Number**	
Remarks:		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1.Custody Seal(s)	Present/Absent*	1			
	Intact/Broken	2			
2.Custody Seal Nos.	N/A	3			
		4			
3.Chain-of-Custody Records	Present/Absent*	5			
		6			
4.Traffic Reports or Packing Lists	Present/Absent*	7			
		8			
5.Airbill	Airbill/Sticker	9			
	Present/Absent*	10			
6.Airbill No.		11			
		12			
7.Sample Tags	Present/Absent*	13			
		14			
Sample Tag Numbers	Listed/ Not Listed on	15			
	Chain-of-Custody	16			
8.Sample Condition	Intact/Broken*/Leaking	17			
		18			
9.Cooler Temperature	°	19			
		20			
10.Does information on custody	Yes/No*	21			
records, traffic reports, and		22			
sample tags agree?		0			
11.Date Received at Lab		24			
		25			
12.Time Received		26			
		27			
Sample Transfer		28			
Fraction	Fraction	29			
Area#	Area#	30			
By	By	31			
On	On	32			
*Contact SMO and attach record of resolution.		33			
Reviewed BY		**Logbook No.**			
Date		**Logbook Page No.**			

Form DC-1 OLM04.2

Attachment 16 (Continued)

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name:				**Page** ___ of ___	
Received By(Print Name)				**Log-in Date**	
Received By(Signature)					
Case Number	**Sample Delivery Group No.**			**Mod. Ref. No.**	
Remarks:			EPA Sample #	Corresponding Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1.Custody Seal(s)	Present/Absent*	1				
	ntact/Broken	2				
2.Custody Seal Nos.		3				
		4				
3.Traffic Reports/	Present/Absent*	5				
Chain of Custody		6				
Records (TR/COCs)		7				
or Packing Lists		8				
4. Airbill	Airbill/Sticker	9				
	Present/Absent*	10				
5. Airbill No.		11				
		12				
6. Sample Tags	Present/Absent*	13				
Sample Tag Numbers	Listed/ Not Listed on	14				
	Chain-of-Custody	15				
7. Sample Condition	Intact/Broken*/Leaking	16				
		17				
8. Cooler Temperature	Present/Absent*	18				
Indicator Bottle		19				
9. Cooler Temperature		20				
		21				
10.Does information on TR/COCs	Yes/No*	22				
and sample tags		23				
agree?		24				
11.Date Received at Laboratory		25				
		26				
12.Time Received		27				
Sample Transfer		28				
Fraction	Fraction	29				
Area#	Area#	30				
By	By	31				
On	On	32				
*Contact SMO and attach record of resolution.		33				
Reviewed BY			**Logbook No.**			
Date			**Logbook Page No.**			

SOM01.1(5/2005)

Attachment 16 (continued)

SAMPLE LOG-IN SHEET

Lab Name:						**Page____of____**	
Received By(Print Name)						**Log-in Date**	
Received By(Signature)							
Case Number	**Sample Delivery Group No.**					**NRAS Number**	
Remarks:			EPA Sample #	Aqueous Sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1.Custody Seal(s)	Present/Absent*	1					
	Intact/Broken	2					
2.Custody Seal Nos.		3					
		4					
3.Traffic	Present/Absent*	5					
Reports/Chain of Custody		6					
Records or Packing Lists	Present/Absent*	7					
4.Airbill	Airbill/Sticker	8					
	Present/Absent*	9					
5.Airbill No.		10					
		11					
6.Sample Tags	Present/Absent*	12					
Sample Tag Numbers	Listed/ Not Listed on	13					
	Traffic Report/Chain	14					
	of Cusody Record	15					
7.Sample Condition	Intact/Broken*/Leaking	16					
		17					
8.Cooler Temperature	Present/Absent*	18					
Indicator Bottle		19					
9.Cooler Temperature		20					
		21					
10.Does information on custody	Yes/No*	22					
records, traffic reports, and		23					
sample tags agree?		24					
11.Date Received at Lab		25					
		26					
12.Time Received		27					
Sample Transfer		28					
Fraction	Fraction	29					
Area#	Area#	30					
By	By	31					
On	On	32					
*Contact SMO and attach record of resolution.		33					
Reviewed by			**Logbook No.**				
Date			**Logbook Page No.**				

Form DC-1 ILM05.3

Attachment 17

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

1. **Client notified laboratory that data would be reported to the State of North Carolina.** _____

2. **Client notified laboratory that data would not be reported to the State of North Carolina.** _____

Dispose - client will resample _____

Project Manager/ID _____/_____ **Date** _____

QAN-R-7
060126

Qanr7 – 1/26/06:jad

Attachment 17 (continued)

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

3. **Client notified laboratory that data would be reported to the State of North Carolina.** _____

4. **Client notified laboratory that data would not be reported to the State of North Carolina.** _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ **Date** _____

Preservation Type_____ **Preservative Lot Number**_____

Preserved By_____ **Date**_____

QAN-R-8
060126

QAN-R-8:012606:jad

Attachment 17 (continued)

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

Client _____
Case # _____
Type of Analysis _____
Receipt Date _____

For some organic and/or inorganic determinations temperature preservation at 4° C is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler. The EPA CLP program requires the laboratory make notification when the temperature exceeds 10° C. The State of North Carolina requires that samples must be iced to above freezing but ≤ 6° C during shipment. Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

1. **Client notified laboratory that data would be reported to the State of North Carolina.** _____

2. **Client notified laboratory that data would not be reported to the State of North Carolina.** _____

Dispose - client will resample _____

Project Manager/ID _____/_____ **Date** _____

QAN-R-9
060309

qanr9 – 3/09/06:jad

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure ✓ revised procedure _____ outdated procedure (archive) _____

- Procedure Code: IP 576 SOP Section #: 3.5.8.3 Revision #: 0

SOP Title: Determination of Hexavalent Chromium in Soil Matrices using SW846 method 7196A

Effective date: (QA fills in) 5/11/06

- Procedure prepared by: V Pospasa Date: 5/10/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Pam Bott New Lab 5/11/06 Date: 5-11-06
- Reason for change: ~~Update~~ New procedure
- This procedure meets the requirements of the following approved method references: "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods" SW-846, 3rd Ed., Update III, 12/96, Method 7196A

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 576: Determination of Hexavalent Chromium in Soil Matrices using SW-846 Method 7196A.

Table of Contents

Instrument Procedure 576: Determination of Hexavalent Chromium in Soil Matrices using SW-846 Method 7196A.

1.0 Scope and Application

This procedure is used to determine the concentration of Hexavalent Chromium [Cr (VI)] in solid matrices prepared using method 3060A.

2.0 Summary of Method

Solid samples are digested according to SOP 3.5.8.2 "Alkaline Digestion of Solid Matrices by Method 3060A for Analysis of Hexavalent Chromium [Cr (VI))]". Diphenylcarbazide solution is added to the resulting sample and the pH of the sample is adjusted with sulfuric acid. The reaction produces a red-violet color. The absorbance of the sample is measured at 540 ηm using a spectrophotometer.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater that zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The reporting limit is based on the MDL, and is usually 4-8 times higher than the MDL.

3.3 Reporting Units – mg/Kg for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and the South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless

the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for the DoD-QSM**,** and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed**.**

3.5 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Substances may interfere with the accurate determination of Cr (VI), if the concentration of chromium is low in the sample being measured

4.2 Hexavalent molybdenum and mercury salts also react with diphenylcarbazide to produce a red-violet color, but the color is much less intense than that produced by the Cr (VI) reaction at the specified pH.

4.3 Vanadium interferes; however, the effects of concentrations of Vanadium up to 10 times that of Cr (VI) are minimal.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Care must be taken when handling corrosive materials such as concentrated acids and bases to prevent injury. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of a failure of the laboratory's ventilation system and must be reported to a supervisor or manager.

Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for information associated with reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Spectrophotometer – Spectronic 21D

6.2 Volumetric pipettes: calibrated automatic pipettes

6.3 Volumetric Flasks: Class A glassware, various sizes, with stoppers, or equivalent.

7.0 Reagents and Standards

7.1 Reagent Water – All water used during preparation must be Type I with regard to resistivity of > 10 megohm-cm at 25° C (20^{th} Editions of Standard Methods, Method 1080).

7.2 Potassium chromate – analytical reagent grade

7.3 Potassium chromate stock solution [1000 mg/L Cr (VI)] – Dissolve 3.731 grams of dried K_2CrO_4 in reagent water in a 1liter flask. Dilute to volume. (One mL of K_2CrO_4 solution equals 1 mg of chromium). Prepare fresh annually.

7.4 Matrix spiking solution – [100mg/L Cr (VI)] – Add 10.0 mL of potassium chromate stock solution (Section 7.3) to a 100 mL volumetric flask and dilute to volume with reagent water. Mix well. Prepare fresh every six months.

7.5 Diphenylcarbazide solution – Dissolve 250 mg of 1, 5-diphenylcarbazide in 50 mL of acetone. Store the solution in an amber bottle. Prepare fresh monthly or sooner, if solution becomes colored.

7.6 Acetone – analytical reagent grade

7.7 Sulfuric acid (H_2SO_4) 10% (v/v) – Add 10mL of distilled reagent grade H_2SO_4 to a 100 mL volumetric flask and dilute to volume with reagent water.

7.8 Five initial calibration standards at concentrations of 0.05, 0.1, 0.4, 1.0, and 2.0 mg/L. See Attachment 1 for standard preparation procedures. Prepare fresh daily.

7.9 Hexavalent Chromium solid reference material – NSI Solutions Inc., Catalog # SQCI-003.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to sample control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples."

8.2 Cr (VI) has been shown to be quantitatively stable in soil field samples for 30 days from collection. Cr (VI) has been shown to be stable in alkaline samples for up to 168 hours (7 days).

8.3 Store digested samples at 4 ºC (± 2º C) until analysis.

9.0 Quality Control

9.1 A method blank must be prepared with each digestion batch. The concentration of Cr (VI) in the method blank must be less that the reporting limit or the batch must be re-digested.

9.2 A laboratory control sample (LCS) must be prepared with each digestion batch. The recovery of Cr (VI) in the LCS must be within 80 to 120 % or the batch must be re-digested.

9.3 A duplicate sample or a matrix spike duplicate must be prepared per SDG. The relative percent difference (RPD) between the duplicate samples must be ≤ 20%, if both the original and duplicate are ≥ 4 times the reporting limit.

9.4 A soluble and insoluble matrix spike must be prepared per SDG. The percent recovery for the matrix spikes should be within 75 to 125%. If the matrix spike recoveries are not with control limits re-digest and analyze samples. If the matrix spike recoveries are still outside of control limits, but the LCS met acceptance criteria report the matrix spikes. Qualify the data in the SDG narrative.

9.5 Verify the absence of reduction reaction and chemical interference that might affect color development by analyzing one post-digestion spike (PDS) per sample matrix.

9.5.1 Prepare the PDS by spiking a 10 mL aliquot of the digested sample. Add enough matrix spiking solution to the sample to double the concentration of Cr (VI) present and analyze.

9.5.2 If the spike recovery in the PDS is within 85 to 115%, the absence of interfering compounds is verified.

9.5.3 If the recovery is outside the control limits, dilute and reanalyze the PDS.

9.5.4 After reanalysis if the recovery of the PDS remains outside of the control limits an alternate determination method should be used.

10.0 Calibration and Standardization

10.1 Verify that a wavelength calibration has been performed on the spectrophotometer within the last two years, before taking any measurements. Refer to SOP 11.3.2.1 "Wavelength Calibration Check for Spectronic 21D Spectrophotometer".

10.2 Develop the color of the standards using the procedure used for the samples. See section 11.0.

10.3 Analyze an initial calibration daily before analyzing any field or QC samples. The standard concentrations analyzed are listed in section 7.8. Measure the absorption at 540 ηm using a 1 cm absorption cell.

10.3.1 Analyze a calibration blank. Correct the absorbance reading for each standard by subtracting the absorbance of the calibration blank.

10.3.2 Analyze the calibration standards. The calibration is acceptable if the correlation coefficient of the curve is ≥ 0.995.

10.3.3 The initial calibration verification (ICV) standard is an LCS prepared from a second source solid reference standard that is carried through the digestion process and analyzed on each analytical run. The recovery of Cr (VI) must be within 90 to 110% of the true value.

10.3.4 Analyze a continuing calibration verification (CCV) standard after every 10 samples and at the end of the analytical run. The control limits for the CCV are 90 to110% of the true value.

11.0 Procedure

Documentation must follow the requirements in QC SOP 13.6 "Proper Documentation Procedures".

11.1 Prior to color development, measure the absorbance of each sample at 540 ηm. Use this value to correct the absorbance of each sample.

11.2 Transfer 95 mL of sample to a 100 mL volumetric flask. Add 2 mL of diphenylcarbazide solution and mix.

11.3 Adjust the pH to between 1.6 and 2.2 with 10% sulfuric acid and dilute to volume with reagent water.

11.4 Let stand for 5 to 10 minutes to allow for full color development.

11.5 Transfer 5 mL of each sample to a 1 cm absorbance cell and measure its absorbance at 540 ηm.

11.6 Analyze a continuing calibration verification (CCV) standard after every 10 analytical samples and at the end of the analytical run.

11.7 Calculate the concentration of Cr (VI) in samples using the equations in section 12.0.

11.8 Results in mg/L are obtained by plotting final absorbance of sample or linear regression of absorbance vs. concentration

12.0 Calculations

Calculations must be consistent with the Quality Control SOP 13.4 "Numerical Data Reduction".

12.1 Calculate concentration of Cr (VI) in (mg/Kg):

$$\text{Conc.} = \frac{\text{Sample concentration (mg/L) x final sample volume (L)}}{\text{Initial sample weight (Kg) x dry weight}}$$

12.2 Calculate the dry weight:

$$\text{dry wt} = \frac{\text{wt container} - \text{dry sample}}{\text{wt container} - \text{wet sample}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\,R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked} \times 100$$

13.0 Method Performance

This method is validated through in-house laboratory studies of method detection limits and precision and accuracy for a single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 7196A.

16.2 Hazardous Waste Management & Safety SOP: Hazardous Waste Disposal" and "Spill Control and Cleanup"

16.3 "Less is Better: Laboratory Chemical Management for Waste Reduction", American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036 (202) 872-4477.

16.4 "The Waste Management Manual for Laboratory Personnel", American Chemical Society

16.5 NELAC Standards, June 2003.

16.6 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.7 CompuChem Quality Manual, Revision 7, Update 1**,** 12/13/05.

17.0 Attachments as Tables, Diagrams and Flow Charts

17.1 Attachment 1 – Hexavalent Chromium Analysis Log

17.2 Attachment 2 – Reagents/Standards Preparation Log for Hexavalent Chromium

Attachment 1

COMPUCHEM a division of Liberty Analytical Corp. LOGBOOK 2 RRRR

Hexavalent Chromium Analysis Log

Modified Method SW-846: 7196A

Analyst _____ Date: _____

SDG ID _____ Correlation Coefficient _____

Reviewed By: _____ Slope: _____

Date: _____ Intercept: _____

#	Sample ID	Acidic pH	Uncorrected Absorbance	Background Absorbance	Corrected Absorbance	Dilution	Concentration	Color	Read Time
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
	CCV								
	CCB								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
	CCV								
	CCB								

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/5/06:jad

Attachment 2

Reagents/Standards Preparation Log for Hexavalent Chromium

CompuChem a division of Liberty Analytical Corp.1 UUUU

NAME	PREPARATION INSTRUCTIONS	PREPARED BY	DATE
Cr (VI) Digestion Solution Ref#:_____	Place in a 1L volumetric flask: 20 g of NaOH (Ref#: _____) 30 g of Na_2CO_3 (Ref#: _____) Dilute to the volume with reagent water. Place solution in a Teflon bottle. Prepare fresh monthly or as needed		
Cr (VI) Stock Standard Solution (1000 mg/L) Ref#:_____	Place in a 1000 mL volumetric flask: 3.731 g K_2CrO_4 (Ref#: _____) Dilute to volume with reagent water. Place solution in a Teflon bottle. Prepare fresh yearly or as needed		
Cr (VI) Intermediate Standard Solution (100 mg/L) Ref#:_____	Place in a 100 mL volumetric flask: 10 mL of Cr (VI) Stock Standard Solution (Ref#: _____) Dilute to volume reagent water. Prepare fresh every six months daily		
2.0 ppm Standard	Place in a 100 mL volumetric flask: 2 ml of Cr (VI) Intermediate Stock Standard Solution (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
1.0 ppm Standard (CCV)	Place in a 100 mL volumetric flask: 1 mL of Cr (VI) Intermediate Standard Solution (Ref#: _____) 50 ml of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.4 ppm Standard	Place in a 100 mL volumetric flask: 0.4 mL of Cr (VI) Intermediate Standard Solution (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.1 ppm Standard	Place in a 100 mL volumetric flask: 0.1 mL of Cr (VI) Intermediate Standard Solution 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.05 ppm Standard	Place in a 100 mL volumetric flask: 0.05 mL of Cr (VI) Intermediate Standard Stock Solution 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
ICB/CCB	50 mL of Cr (VI) Digestion solution (Ref#: _____)		

Page 1 of 2

Attachment 2 (continued)

Reagents/Standards Preparation Log for Hexavalent Chromium

CompuChem a division of Liberty Analytical Corp.1 UUUU

NAME	PREPARATION INSTRUCTIONS	PREPARED BY	DATE
ICV	Place in a 250 mL beaker: 2.5 g of Hexavalent Chromium reference standard (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____)		
Matrix Spiking Solution Ref#:_____	Place in a 1 mL volumetric flask: 10 mL of Cr (VI) Stock Standard Solution (Ref#: _____) Dilute to volume with rengent water		
Phosphate Buffer Solution Ref#:_____	Place in 1 L volumetric flask: 87.09g of K_2HPO_4 (Ref#: _____) and 68.04 g of KH_2PO_4 (Ref#: _____) Dilute to volume with rengent water. Prepare fresh every six months		
Diphenylcarbazide Solution Ref#:_____	Place in a 50 mL volumetric flask: 0.25 g of 1,5-diphenylcarbazide Dilute to volume with acetone. Prepare fresh monthly of sooner, if solution becomes colored.		

Reviewed by: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/11/06 jad

Page 2 of 2

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Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure ✓ revised procedure _____ outdated procedure (archive) _____

- Procedure Code: SPP-222 SOP Section #: 3.5.8.3 (Rev 5/11/06 2) Revision #: 0

SOP Title: Alkaline Digestion of Solid Matrices by Method 3060A for analysis of Hexavalent Chromium

Effective date: (QA fills in) 5/11/06

- Procedure prepared by: Reapass Date: 5/11/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 5-11-06
- Reason for change: New SOP 5/11/06 Updated Procedure
- This procedure meets the requirements of the following approved method references: "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd edition, Update III, 12/96, Method 3060A

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

ORIGINAL

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -222: Alkaline Digestion of Solid Matrices by Method 3060A for Analysis of Hexavalent Chromium [Cr (VI))]

Table of Contents

Sample Preparation Procedure -222: Alkaline Digestion of Solid Matrices by Method 3060A for Analysis of Hexavalent Chromium [Cr (VI))]

1.0 Scope & Application

This procedure is used to prepare solid (soils, sediment, sludge, waste) samples for determination of Cr (VI) by colorimetric method 7196A.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Samples are digested in a hot basic solution to solubilize both water-soluble and water-insoluble Cr (VI) in solid matrices.

3.0 Definitions

3.1 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case,
- or each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and the South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must

also be prepared together at a rate of 5% for the DoD-QSM, and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.2 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

The oxidation of native Cr (III) contained in the sample to Cr (VI) or the reduction of native Cr (VI) to Cr (III) will interfere with the accurate determination of Cr (VI). Under alkaline conditions minimal oxidation of Cr (III) or reduction of Cr (VI) occur.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Care must be taken when handling corrosive materials such as concentrated acids and bases to prevent injury. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of a failure of the laboratory's ventilation system and must be reported to a supervisor or manager.

Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for information associated with reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Digestion vessel –250 mL Pyrex® beaker

6.2 Graduated cylinders – 100 mL or equivalent.

6.3 Volumetric Flasks – Class A glassware, 1000 mL and 100 mL, with stoppers, or equivalent.

6.4 Vacuum Filtration Apparatus – Corning® 115 mL Filter System, Model # 430944

6.5 Filter paper – 0.45 µm.

6.6 Mini Hot plate/stirrer – VWR 220

6.7 Volumetric pipettes – calibrated automatic pipettes.

6.8 pH meter – Fisher Scientific pH meter 50

6.9 Balance – Denver Instrument Company, Model# 00070

6.10 Thermometer – NIST traceable, alcohol

6.11 Magnetic stir bars

7.0 Reagents & Standards

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080).

7.2 Nitric acid (HNO_3) – 5.0 M, analytical reagent grade

7.3 Sodium carbonate (Na_2CO_3) – anhydrous, analytical reagent grade

7.4 Sodium Hydroxide (NaOH) – analytical, reagent grade

7.5 Potassium chromate (K_2CrO_4) – analytical reagent grade

7.6 Magnesium Chloride ($MgCl_2$) – anhydrous, analytical reagent grade

7.7 Digestion solution – Dissolve 20.0 grams of NaOH and 30.0 grams of Na_2CO_3 in reagent water in a 1 liter flask. Dilute to volume, and store in a tightly capped polyethylene bottle. Prepare digestion solution fresh monthly.

Note: Check the pH of the digestion solution prior to each use. The pH must be >11.5.

7.8 Potassium chromate stock solution [1000 mg/L Cr (VI)] –Dissolve 3.731 grams of dried K_2CrO_4 in reagent water in a 1 liter flask. Dilute to volume. (One mL of K_2CrO_4 solution equals 1 mg of chromium). Prepare fresh annually.

7.9 Matrix spiking solution – [100mg/L Cr (VI)] – Add 10.0 mL of potassium chromate stock solution (Section 7.8) to a 100 mL volumetric flask and dilute to volume with reagent water. Mix well. Prepare fresh every six months.

7.10 Lead Chromate (PbCrO4) – analytical reagent grade; store under dry conditions at 20 - 25° C in a tightly sealed container.

7.11 Hexavalent Chromium solid reference material – NSI Solutions Inc., Catalog # SQCI-003

7.12 Phosphate buffer:

7.12.1 K_2HPO_4 – analytical grade

7.12.2 KH_2PO_4 – analytical grade

7.12.3 Prepare the phosphate buffer solution by dissolving 0.5 M K_2HPO_4 (87.09 g) and 0.5 M KH_2PO_4 (68.04 g) into 700 mL of reagent water. Transfer to a 1 liter volumetric flask and dilute to volume. Prepare fresh every six months.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed."

8.2 Field samples are stored at 4° C (± 2° C) prior to preparation.

8.3 Cr (VI) has been shown to be quantitatively stable in soil field samples for 30 days from collection. Cr (VI) has been shown to be stable in alkaline samples for up to 168 hours (7 days).

8.4 Store samples at 4° C (± 2° C) until analysis.

9.0 Quality Control

All quality control samples in this section are processed in the same manner as the field samples. (See section 11.0.)

9.1 Prepare a method blank with each digestion batch.

9.1.1 Prepare a solid method blank by weighing 2.5 g (± 0.1 g) of blank sand into a digestion vessel.

9.2 Prepare a laboratory control sample (LCS) with each digestion batch.

9.2.1 Prepare the solid matrix spike by weighing 2.5 g (± 0.1 g) of Hexavalent Chromium solid reference material into a digestion vessel.

9.3 Prepare one duplicate sample or matrix spike duplicate per SDG.

9.4 Prepare one soluble and one insoluble matrix spike per SDG.

9.4.1 Prepare the soluble matrix spike by adding 40 mg/Kg of matrix spike solution (section 7.9) or 2 times the sample concentration, whichever is greater, to the selected sample.

9.4.2 Prepare the insoluble matrix spike by adding 10 to 20 mg of Lead Chromate to a separate sample.

10.0 Calibration and Standardization

10.1 Calibrate the analytical balance using reference weights prior to use.

10.2 Calibrate automatic pipettes monthly.

10.3 Calibrate the thermometers annually, using a NIST traceable thermometer.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 Remove any sticks or rocks from the sample container and thoroughly mix the sample before removing an aliquot.

11.2 Place 2.5 g (± 0.1 g) of sample into a clean labeled digestion vessel. Place a magnetic stir bar into the vessel. Add spike to the matrix spikes.

11.3 Add 50 mL of digestion solution to each sample, using a graduated cylinder. Also add 400 mg of $MgCl_2$ and 0.5 mL of phosphate buffer solution to each sample. (For analytical techniques that can correct for oxidation/reduction of chromium, the addition of $MgCl_2$ is optional). Cover all samples with watch-glasses.

11.4 Stir the samples continuously unheated for 5 minutes.

11.5 Heat the samples to 90 – 95° C. Maintain the samples at 90 – 95° C for 60 minutes, stirring constantly.

11.5.1 Monitor the digestion temperature using a temperature blank. Prepare the temperature blank by placing a thermometer into a digestion vessel containing 50 mL of digestion solution.

11.6 Cool the samples to room temperature, stirring constantly.

11.7 Quantitatively transfer the contents of the digestion vessel to a vacuum filtration apparatus, rinsing the vessel 3 times with reagent water. Filter through a 0.45 µm filter paper.

11.8 Rinse the filter flask and filter paper with reagent water and transfer contents to a clean digestion vessel.

11.9 Add a stir bar to the vessel and place on a stirrer. SLOWLY add 5.0 M HNO_3 drop wise to the sample. Perform this step in the fume hood.

11.10 Adjust the pH of the sample to between 7.0 and 8.0. If the pH falls outside the range, discard and re-digest the sample.

11.11 Remove stirrer and quantitatively transfer the sample to a 100 mL volumetric flask. Dilute to volume with reagent water.

11.12 Analyze samples using SOP "Determination of Hexavalent Chromium in Soil Matrices using SW-846 Method 7176A".

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4 "Numerical Data Reduction".

13.0 Method Performance

This method is validated through in-house laboratory studies of method detection limits and precision and accuracy of a single analyst. The data are retained in the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has

established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3060A.

16.2 "Less is Better: Laboratory Chemical Management for Waste Reduction", American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036 (202) 872-4477.

16.3 NELAC Standards, June 2003.

16.4 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.5 CompuChem Quality Manual, Revision 7, Update 1, 12/13/05.

17.0 Attachments as Tables , Diagrams, and Flowcharts

17.1 Attachment 1 – Hexavalent Chromium Digestion log

17.2 Attachment 2 – Reagents/Standards Preparation Log for Hexavalent Chromium

Attachment 1

CompuChem, a division of Liberty Analytical Corp.

Worksheet 1 J (8) **Cr(VI) DIGESTION PREP** Page __1___ of __1___

SDG # _____ **SW-846 Method 3060A**

Analyst No.: _____

CompuChem No.	Sample Type	Sample weight (gms)	Hot Plate Temp °C	Sample filtered? (Y or N)	Final volume (mL)	Comments

Digestion Solution - _____ - _____

Phosphate Buffer Solution - _____ - _____

Digestion Time Start - _____

Matrix Spiking Solution (100mg/L CrVI) - _____ - _____

Digestion Time Stop - _____

NaOH ref#	
K_2HPO_4 ref#	
KH_2PO_4 ref#	

Reviewed By: _____ Date: _____

The presence of the Chemist's/analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/09/06:jad

Attachment 2

Reagents/Standards Preparation Log for Hexavalent Chromium

CompuChem a division of Liberty Analytical Corp.1 UUUU

NAME	PREPARATION INSTRUCTIONS	PREPARED BY	DATE
Cr (VI) Digestion Solution Ref#:_____	Place in a 1L volumetric flask: 20 g of NaOH (Ref#: _____) 30 g of Na_2CO_3 (Ref#: _____) Dilute to the volume with reagent water. Place solution in a Teflon bottle. Prepare fresh monthly or as needed		
Cr (VI) Stock Standard Solution (1000 mg/L) Ref#:_____	Place in a 1000 mL volumetric flask: 3.731 g K_2CrO_4 (Ref#: _____) Dilute to volume with reagent water. Place solution in a Teflon bottle. Prepare fresh yearly or as needed		
Cr (VI) Intermediate Standard Solution (100 mg/L) Ref#:_____	Place in a 100 mL volumetric flask: 10 mL of Cr (VI) Stock Standard Solution (Ref#: _____) Dilute to volume reagent water. Prepare fresh every six months daily		
2.0 ppm Standard	Place in a 100 mL volumetric flask: 2 ml of Cr (VI) Intermediate Stock Standard Solution (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
1.0 ppm Standard (CCV)	Place in a 100 mL volumetric flask: 1 mL of Cr (VI) Intermediate Standard Solution (Ref#: _____) 50 ml of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.4 ppm Standard	Place in a 100 mL volumetric flask: 0.4 mL of Cr (VI) Intermediate Standard Solution (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.1 ppm Standard	Place in a 100 mL volumetric flask: 0.1 mL of Cr (VI) Intermediate Standard Solution 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
0.05 ppm Standard	Place in a 100 mL volumetric flask: 0.05 mL of Cr (VI) Intermediate Standard Stock Solution 50 mL of Cr (VI) Digestion solution (Ref#: _____) Dilute to volume with reagent water. Prepare fresh daily		
ICB/CCB	50 mL of Cr (VI) Digestion solution (Ref#: _____)		

Page 1 of 2

Attachment 2 (continued)

Reagents/Standards Preparation Log for Hexavalent Chromium

CompuChem a division of Liberty Analytical Corp.1 UUUU

NAME	PREPARATION INSTRUCTIONS	PREPARED BY	DATE
ICV	Place in a 250 mL beaker: 2.5 g of Hexavalent Chromium reference standard (Ref#: _____) 50 mL of Cr (VI) Digestion solution (Ref#: _____)		
Matrix Spiking Solution Ref#:_____	Place in a 1 mL volumetric flask: 10 mL of Cr (VI) Stock Standard Solution (Ref#: _____) Dilute to volume with reagent water		
Phosphate Buffer Solution Ref#:_____	Place in a 1 L volumetric flask: 87.09g of K_2HPO_4 (Ref#: _____) and 68.04 g of KH_2PO_4 (Ref#: _____) Dilute to volume with reagent water. Prepare fresh every six months		
Diphenylcarbazide Solution Ref#:_____	Place in a 50 mL volumetric flask: 0.25 g of 1,5-diphenylcarbazide Dilute to volume with acetone. Prepare fresh monthly of sooner, if solution becomes colored.		

Reviewed by: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/11/06 jsd

Page 2 of 2

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP564 SOP Section #: 3.5.8.1 Revision #: 5

SOP Title: Colorimetric Determination of Hexavalent Chromium in Aqueous Samples by Lachat, Standard Methods, and SW-846

Effective date: (QA fills in) 5/11/06

- Procedure prepared by: Wespac2 Date: 5/1/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 5-11-06
- Reason for change: update procedure
- This procedure meets the requirements of the following approved method references: Lachat QuikChem AE Method 10-124-13-1-A, 7/86; 19th Ed. of Standard Methods, 1995, Method 3500-Cr-D; 20th Ed. of Standard Methods, 1998 Method Cr-B Test Methods for Evaluating Solid Waste: Physical/Chemical Methods SW-846 3rd Edition, Update III, 12/96; Method 7196A

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 564: Colorimetric Determination of Hexavalent Chromium in Aqueous Samples by Lachat, Standard Methods, and SW-846

Table of Contents

Instrument Procedure 564: Colorimetric Determination of Hexavalent Chromium in Aqueous Samples by Lachat, Standard Methods, and SW-846

1.0 Scope and Application

This automated colorimetric method is suitable for ground water, surface waters, domestic and industrial wastewaters, and leachate extracts and digestates. This procedures measures only dissolved hexavalent chromium (Cr VI). The applicable range is 10 – 400 μg/L.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP's requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. This reaction produces a red violet color with a maximum absorbance at 540 ηm.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For some inorganic methods, the reporting limit is based on the MDL, and is usually 4-8 times higher than the MDL. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.3 Reporting Units – μg/L

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD-QSM** and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for **DoD-QSM** and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

3.6 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Substances that can reduce CR (VI) upon acidification such as cyanide and thiosulfate will cause negative interference in the results.

4.2 Hexavalent molybdenum and mercury salts react to form color with the reagent, however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Vanadium interferes strongly is present at concentrations above 10 times that of chromium.

4.3 Iron concentrations greater than 1 mg/L may produce a yellow color, bu thte ferric iron color is not strong and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are **required** to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 **Zellweger Analytics** Lachat QuikChem® 8000 Series **FIA System**

6.1.1 Automatic sampler

6.1.2 Proportioning pump

6.1.3 Injection manifold

6.2 Magnetic stirrer

6.3 Various glassware including volumetric flasks

6.4 Corning® 115 mL Filter System, Mode #430944

7.0 Reagents and Standards

7.1 Carrier Solution: Reagent Water - All water used during preparation **is** reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080.

7.2 Potassium chromate (K_2CrO_4) – analytical, reagent grade

7.3 Color Reagent - **Diphenylcarbazide solution**

7.**3**.1 Dissolve 0.20g of diphenylcarbazide in 100 mL of isopropanol in a 500 mL volumetric flask. Stir with a magnetic stirrer until dissolved. Add approximately 350 mL **reagent water** and 40 mL concentrated H_2SO_4 and dilute to **volume** with **reagent water**. Mix with a magnetic stirrer. This solution is stable for one month.

7.3.2 Prepare fresh monthly or when solution becomes colored.

7.4 Sulfuric acid (H_2SO_4) – analytical, reagent grade

7.**5** **Cr(VI) Stock Solution A (100 ppm) – D**issolve **0.3735** g dried potassium chromate in 1 L **reagent water**. **Prepare fresh annually.**

7.6 Cr(VI) Stock B (100 ppm) – prepared as in Section 7.5 from an alternate source or lot. Prepare fresh annually.

7.7 Cr(VI) Intermediate Standard A (10 ppm) – Add 10 mL of Cr(VI) stock solution A to a 100mL volumetric flask and dilute to volume. Prepare fresh every six months.

7.**8** **Cr(VI) ICV Solution (250 ppb) – Add 0.25mL of Cr(VI) stock B to a 100 mL volumetric flask and dilute to volume**. **Prepare fresh every six months.**

7.**9** **Calibration** Standards **–** Add 4.0, 2.0, 1.0, 0.5, 0.2, **and** 0.1 **mLs** of the **Cr(VI)** intermediate **standard A** to **six** 100 mL volumetric flasks. Dilute **each to volume with reagent water, resulting in concentrations of** 400, 200, 100, 50, 10 µg/L**, respectively**.

7.10 Calibration Blanks (ICB/CCB) – Reagent water analyzed after the initial calibration and the continuing calibration verifications.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and SOP 4.6, "Storing Samples."

8.**2** **Store** sample**s** at 4° C and run as soon as possible**,** but within 24 hours of sampling.

8.**3** The holding time for this test is 24 hours from the time of collection.

9.0 Quality Control

9.1 Initial Calibration Verification (ICV) **–** the ICV solution is a 250 µg/L solution prepared from a separate lot source of potassium chromate.

9.1.1 The result of the ICV must agree within 90 - 110% of the true value. The true value is 250 µg/L. When the ICV is outside this range, the system must be checked, corrected, and recalibrated. Limits must be met before sample analysis can proceed.

9.2 Initial Calibration Blank (ICB) **–** A reagent blank is used to establish that the **instrument is** free of contamination. One ICB must be analyzed per **analytical sequence**.

9.2.1 The result of the ICB must not be greater than the reporting limit. If the limit is exceeded, determine and correct the problem and recalibrate.

9.3 Continuing Calibration Verification (CCV) **–** The CCV is **the** 200 µg/L **calibration** standard.

9.3.1 The result of the CCV must agree within 90-110% of the true value. The true value is 200 µg/L. The CCV must be analyzed every 10 analytical samples. When the CCV is out of range, determine and correct the problem, recalibrate, and reanalyze all affected samples.

9.4 Continuing Calibration Blank (CCB) **–** The CCB is a reagent blank used to establish that the **instrument is** free from contamination. A CCB **is** analyzed every 10 analytical samples.

9.4.1 The result of the CCB must not be greater than the reporting limit. If the **result exceeds the reporting**, determine and correct the problem, recalibrate, and reanalyze the affected samples.

9.5 Method Blank **– The method blank is reagent water prepared in the same manner as the samples. It** is used to establish that the reagents and glassware are free from contamination. One method blank must be analyzed per every twenty samples. For SC DHEC analyzed the blank every 10 samples. The method blank must be identified on the run log.

9.5.1 If any value present in the method blank exceeds the reporting limit, the entire batch must be reprocessed using clean glassware.

9.6 Laboratory Control Sample (LCS) **–** The LCS is a 250 µg/L solution prepared from a separate lot source of potassium chromate. The LCS must be prepared with every 20 samples or each SDG, whichever is more frequent. For SC DHEC**,** analyze **an LCS with** every 10 samples. Accuracy of the process is determined from the LCS recovery.

9.6.1 The result of the LCS must agree within 90 - 110% of the true value. The true value is 250 µg/L. When the LCS is outside this range, the system must be checked, a new QC sample made up, and the associated batch of samples must be reprocessed.

9.7 Matrix Spike and Matrix Spike Duplicate (MS/MSD) **–** Two additional aliquots of native sample are spiked with 1.0 mL of 10 mg/L intermediate standard

solution to yield a spike concentration of 100 µg/L. The MS/MSD pair is prepared for every SDG. Precision and accuracy are determined from the MS/MSD RPD. Failures must be documented on the runlog.

9.7.1 For SC DHEC a duplicate is required at a 10% frequency. The MS/MSD count towards this requirement.

9.7.2 The MS/MSD recovery **should** fall within 75-125% of expected and the RPD **should** be < 20%. If the MS/MSD fail criteria but the LCS passes, the results may be reported with the failure attributed to the sample matrix. If a spiking error can be identified, the MS/MSD may be reprepared, or the amount of spike adjusted in the case of known double-spiking.

9.8 Contingency

9.8.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.8.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.8.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 Perform an initial calibration at the beginning of each sequence and when the CCV fails. See Procedure (11.0) below for steps in the calibration.

10.2 Calibration curve linearity: After the instrument has been calibrated and before any samples can be analyzed, the correlation coefficient must be determined to be 0.995 or greater.

11.0 Procedure

Documentation must follow the requirements in Q**uality** C**ontrol** SOP **13.6 "**Proper Documentation Procedure**"**.

11.1 Filter each sample and a method blank prior to analysis.

11.2 Perform an initial calibration by analyzing a 10 mL aliquot of each calibration standard. Analyze an ICB after the six calibration standards.

11.3 Analyze a CCV and CCB after every 10 samples and at the end of the analytical sequence.

11.4 If the concentration of Cr(VI) exceeds the concentration of the high calibration standard, dilute and reanalyze the sample.

11.5 Operating the Lachat QuikChem 8000 Series

11.**5**.1 Inspect the manifold for proper connections.

11.**5**.2 Turn on the power to the instrument.

11.**5**.3 Secure the pump cassettes and place transmission lines in the carrier and color reagent.

11.**5**.4 Select and download the method for hexavalent chromium.

11.**5**.5 Allow the system to pump until the baseline is steady.

11.**5**.6 Place the standards in the rack in descending order of concentration followed by QC and samples following the sequence outlined in the Hexavalent Chromium Run Log.

11.**5**.7 Enter the sample IDs into the sample tray.

11.**5**.8 Submit the tray and calibrate the instrument.

11.**5**.9 When the tray is complete, rinse the lines with **reagent water**. Pump water through the system for 10 minutes to remove any reagents in the lines.

11.**5**.10 Once lines are rinsed, remove the carrier and reagent lines from the water and pump air through the system until it is dry.

11.**5**.11 To release tension on the pump tubing, remove pump cassettes and turn off the instrument.

12.0 Data Analysis and Calculations

All calculations must be consistent with the Q**uality** C**ontrol** SOP **13.4 "**Numerical Data Reduction**"**.

12.1 Calculate concentration of hexavalent chromium (µg/L):

Concentration = (sample conc. µg/L) x (dilution factor)

12.2 Calculate **dry weight**:

$$\textbf{dry wt} = \frac{\text{wt container – dry sample}}{\text{wt container – wet sample}}$$

12.3 Calculate concentration of Cr(VI) in a water leachate (mg/Kg)

$$\textbf{Conc.} = \frac{\textbf{(Sample concentration in µg/L)(Final sample volume in L)}}{\textbf{(Dry weight)(Sample weight in g)}}$$

12.**4** Percent Recovery (%R):

$$\textbf{\%R} = \frac{\text{SSR – SR x 100}}{\text{SA}}$$

where:
SSR = Sample Spike Result
SR = Sample Result
SA = Spike Added

12.**5** Relative Percent Difference (RPD):

$$\text{RPD} = \frac{\textbf{(}\text{MSR - MSDR}\textbf{)} \text{ x 100}}{\text{½ (MSR = MSDR)}}$$

where: MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

12.**6** Data Review and Verification

12.**6**.1 Review and verification must follow the procedures in the Inorganic Wet Chemistry and Organic Characterization SOP 14.3.2.1, "Procedures for Wet Chemistry Data Review and Verification".

12.**6**.2 Initial review is performed by the analyst who also generates the final report. A peer or supervisory review is performed prior to release of analytical results to the client.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. **See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.**

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Lachat QuikChem Method 10-124-13-1-A, July 1986

16.2 19th Edition of Standard Methods, 1995, Method 3500 Cr-D and 20th Edition of Standard Methods, 1998, Method 3500 Cr-B.

16.3 Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Method 7196A

16.4 "Less is Better: Laboratory Chemical Management for Waste Reduction", American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036 (202) 872-4477.

16.5 NELAC Standards, June 2003, plus revisions

16.6 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.7 CompuChem Quality Manual, Revision 7, Update 1, 12/13/05, with revisions

17.0 Attachments as Tables, Diagrams, **&** Flowcharts

17.1 Attachment 1 - Hexavalent Chromium Analysis Run Log Example

17.2 Attachment 2 - QC/Standards Preparation Log

Attachment 1

Hexavalent Chromium Run Log 1

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Analyst:_____ File Name(s):_____

Date:_____ Case Name(s):_____

Instrument ID: C2 _____

Note: Manual Dilution Factor is not reflected in the sample resu Lachat Method 10-124-13-'-A
Method SW846 7196A

Cup Number	Sample ID	Sampling Date	Sampling Time	Manual Dil Factor	Result/Peak Area	Unit	Analyte Name	Comments
1	S400	14-Apr-06	11:29:52	1	3208579	uv-a	Hexavalent Chromium	
2	S200	14-Apr-06	11:30:52	1	1723245	uv-a	Hexavalent Chromium	
3	S100	14-Apr-06	11:31:51	1	881101	uv-a	Hexavalent Chromium	
4	S50	14-Apr-06	11:32:51	1	486778	uv-a	Hexavalent Chromium	
5	S20	14-Apr-06	11:33:51	1	196493	uv-a	Hexavalent Chromium	
6	S10	14-Apr-06	11:34:50	1	82125	uv-a	Hexavalent Chromium	
7	S0.0	14-Apr-06	11:35:50	1	0	uv-a	Hexavalent Chromium	
13	ICV	14-Apr-06	11:37:50	1	247.138	µg/L	Hexavalent Chromium	
14	ICB	14-Apr-06	11:38:51	1	-3.52334	µg/L	Hexavalent Chromium	
1	PBW	14-Apr-06	11:40:51	1	-2.826777	µg/L	Hexavalent Chromium	
2	LCSW	14-Apr-06	11:41:52	1	249.374252	µg/L	Hexavalent Chromium	
3	MH2-600-0406	14-Apr-06	11:42:52	1	-1.617853	µg/L	Hexavalent Chromium	
4	MH2-MS	14-Apr-06	11:43:51	1	102.94175	µg/L	Hexavalent Chromium	
5	MH2-MSD	14-Apr-06	11:44:51	1	102.952065	µg/L	Hexavalent Chromium	
6	MH2-DUP	14-Apr-06	11:45:50	1	-1.529416	µg/L	Hexavalent Chromium	
2	CCV	14-Apr-06	11:46:50	1	213.053986	µg/L	Hexavalent Chromium	
7	CCB	14-Apr-06	11:47:51	1	-4.943047	µg/L	Hexavalent Chromium	

Reviewed by:_____ Date:_____

Attachment 2

Standards/QC Preparation for Chromium (VI) on Lachat

Seq. File Name: _____

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 XXX

STANDARDS AND REAGENTS TRACEABILITY*	Ref#
Potassium Chromate, dried (used in stock solution A)	7M2-270-6
Potassium Chromate, dried (used in standard B)	7M2-227-8
Concentrated H_2SO_4	1M2-165-6
Isopropanol	1M2-157-13
Diphenylcarbohydrazide	7M2-229-16
ITEM TO BE PREPARED**	**PREPARATION INSTRUCTIONS**
Color Reagent – 1 1 2 6 0 5 – J M	Dissolve 0.20g of diphenylcarbazide in 100 mL of isopropanol in a 500 mL volumetric flask. Stir with a magnetic stirrer until dissolved. Add approximately 350 mL DI H_2O and 40 mL concentrated H_2SO_4 and dilute to the line with DI H_2O. Mix with a magnetic stirrer. This solution is stable for one month. Note: This solution will gradually change from colorless to tan. This will not affect its use.
Chromium (VI) stock solution A (100 ppm) – 0 5 0 8 0 5 – J M	Place in a 1000-ml volumetric flask: 0.3735 g of dried potassium chromate Dilute to the mark with deionized water. Prepare fresh yearly.
Chromium (VI) standard B (100 ppm) – 0 5 0 8 0 5 – J M	Place in a 1000-ml volumetric flask: 0.3735 g ml of dried potassium chromate from second source Dilute to mark with deionized water. Prepare fresh yearly.
Chromium (VI) Intermd. Std. A (10 ppm) - 0 6 1 4 0 5 – J M	Place into a 100-ml volumetric flask: 10 ml of chromium (VI) stock solution A Dilute to mark with deionized water. Prepare fresh every six months
Chromium (VI) standard (400 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 4.0 ml of chromium (VI) stock intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed.
Chromium (VI) standard (200 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 2.0 ml of chromium (VI) intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed.
Chromium (VI) standard (100 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 1.0 ml of chromium (VI) intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed
Chromium (VI) standard (50 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 0.5 ml of chromium (VI) intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed
Chromium (VI) standard (20 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 0.5 ml of chromium (VI) intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed
Chromium (VI) standard (10 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 0.1 ml of chromium (VI) intermediate standard A Dilute to mark with deionized water. Prepare fresh weekly or more often as needed
Chromium (VI) ICV Solution (250 ppb) – 1 1 2 6 0 5 – J M	Place in a 100-ml volumetric flask: 0.25 ml of 100 ppm chromium (VI) standard B Dilute to mark with deionized water. Prepare fresh weekly or more often as needed

Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log **Standard Lot Number consists of standard ID, date prepared, and initials of preparer. 1/20/06:jad

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

CompuChem
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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) 1/11/06

- Procedure Code: IP 930 SOP Section #: 3.4.5 Revision #: 6

SOP Title: Cyanide Analysis of Water and Soil/Sediment Distillates by CLP, mcAWW, SW-846, NYSASP, and Lachat

Effective date: (QA fills in) 1/11/06

- Procedure prepared by: _____ Date: 01-10-06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) _____ Date: 1-10-06
- Reason for change: Annual review and response to EPA audit
- This procedure meets the requirements of the following approved method references: US EPA CLP SOW ILM04.1, ILM05.3, 335.2/335.3 (CLP-M); SW-846, 3rd Edition, Update III, Method 9010B and Method 9012A; mcAWW, March 1983, Method 335.2 and Method 335.3

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

_____ _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 930: Cyanide Analysis of Water and Soil/Sediment Distillates by CLP, MCAWW, SW-846, NYSASP, and Lachat

Table of Contents

Instrument Procedure 930: Cyanide Analysis of Water and Soil/Sediment Distillates by CLP, MCAWW, SW-846, NYSASP, and Lachat

1.0 Scope and Application

This procedure is applicable to the determination of total and free cyanide in water wastes, including soils/sediments, at concentrations of 10 ppb-200 ppb for CLP, 335.2/335.3 (CLP-M), MCAWW and 10ppb-400 ppb for **SW-846**. The method detects inorganic cyanides that are present as either soluble salts or complexes. The procedure for calculating cyanide amenable to chlorination is also included.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

Total cyanide from alkaline distillates of water and soil/sediment is converted to cyanogen chloride, CNCl, by reaction with choramine-T at pH lower than 8. The CNC1 then forms a red-blue dye reacting with pyridine-barbituric acid reagent. The color is read at 570 **η**m. The concentration of NaOH must be the same in the standards, the scrubber solutions, and any dilution of the original scrubber solutions to obtain colors of comparable intensity.

Prior to analysis samples are distilled according the Sample Preparation Procedure –072, "Aqueous Sample Midi-Distillation by CLP and NYSASP," Sample Preparation Procedure –093, "Aqueous Sample Total and Free Cyanide Midi-Distillation by **SW-846** and NYSASP," Sample Preparation Procedure –139, "Solid Sample for Total Cyanide Midi-Distillation by CLP and NYSASP," or Sample Preparation Procedure –191, "Solid Sample for Total and Free Cyanide Midi-Distillation by SW-846 and NYSASP."

Amendable cyanide is calculated by subtracting the amount of cyanide determined in the pre-treated sample (using Sample Preparation Procedure –094, "Midi Distillation of Aqueous Samples for Amenable Cyanide by CLP and NYSASP" or following Sample Preparation Procedure –245: Cyanide Extraction Procedure for Solids and Oils by **SW-846**") from the amount of total cyanide determined from the untreated sample distillate.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For some inorganic methods, the reporting limit is based on the MDL, and is usually 4 - 8 times higher than the MDL.

3.3 Reporting Units – (will vary with the method)

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers (US ACE) and SC DHEC does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for US ACE and SC DHEC soils The rate is 10% for SC DHEC water samples. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

3.6 For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides, fatty acids, aldehydes and thiocyanates may distill over.

4.0 Interferences

4.1 For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides, fatty acids, aldehydes and thiocyanates may distill over.

5.0 Safety

5.1 Always wear the proper safety equipment (labcoat, safety glasses, and gloves) when performing this procedure.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Cyanide manifold 10.204.00.1.A

6.2 Heating unit

6.3 Vacuum pump

7.0 Reagents & Standards

Note: Standard preparations and reagent lot numbers are documented on the Standards/QC Preparation for Cyanide Analysis on Lachat log (Attachment 2).

Note: To prevent bubble formation, degas all solutions except the standards using a vacuum pump.

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (**20**th Edition of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Reagent 1 - Carrier, 0.25M NaOH

7.2.1 In a 1 L volumetric flask, dissolve 10.0 g of NaOH in 800 mL of water. Dilute to the mark and invert three times.

7.3 Reagent 2 - Phosphate Buffer (0.71 M)

7.3.1 In a 1 L volumetric flask, dissolve 97 g of potassium phosphate monobasic anhydrous (KH_2PO_4) in 800 mL of water. Dilute to the mark and invert three times.

7.4 Reagent 3 - Chloramine-T

7.4.1 To a 500 mL volumetric flask, add 250 mL of water followed by 2 g of chloramine-T. Dilute to the mark and invert three times. Prepare fresh daily.

7.5 Reagent 4 - Pyridine-Barbituric Acid

CAUTION - Always use a fume hood when preparing this reagent. Always wear a face shield when dispensing concentrated acids. This reagent has a **six-**month shelf - life if stored at 4 °C.

7.5.1 In the fume hood, place 15.0 g of barbituric acid in a 1 L beaker and add 100 mL of water, rinsing down the sides of the beaker to wet the barbituric acid. Stir in 75 mL of pyridine (C_5H_5N) and mix until the barbituric acid dissolves. Add 15 mL of concentrated hydrochloric acid (12M HC1) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert three times.

7.6 Stock Standard at 1000.0 mg CN-/L**: purchase 1000 ppm cyanide standard from NSI or equivalent supplier.**

7.7 Intermediate Standard at 10.0 mg CN-/L

7.7.1 Pipet 1.0 m**L** of the Stock Standard into a 100 **mL**volumetric flask. Dilute to the mark with Reagent 1. Invert three times. Prepare with each day of sample analysis.

7.8 Calibration Curve

7.8.1 Five working standards are prepared daily when samples are analyzed at 200, 100, 50, 20, 10 μg CN-/L for CLP**, (ILM05.3) CRI**.

7.8.1.1 To five 100 **mL** volumetric flasks add, respectively, 2.0, 1.0, 0.5, 0.2, and 0.1 **mL** of the 10 mg/L intermediate standard. Dilute to the mark with Reagent 1 and invert at least three times. A zero standard is used which contains only Reagent 1.

7.8.1.2 The 100 **μ**g CN-/L standard is utilized as the continuing calibration verification (CCV).

7.8.1.3 For ILM05.3 the 10 μg CN-/L standard is utilized as the CRI.

7.8.2 Six working standards are prepared daily when samples are analyzed at 400, 300, 200, 100, 40, 20, 10 **μ**g/L for SW-846.

7.8.2.1 To six 100 **mL** volumetric flasks add, respectively, 4.0, 3.0, 2.0, 1.0, 0.4, 0.2, and 0.1 **mL** of the 10 mg/L intermediate standard. Dilute to the mark with Reagent 1 and invert at least three times. A zero standard is used which contains only Reagent 1. **This is also the ICB/CCB.**

7.9 A duplicate must be analyzed from each group of samples of a similar matrix (up to 20 samples) or for each SDG.

A control limit of 20% for RPD shall be used for values greater than or equal to 5x CRDL/**CRQL** reporting limit or ± CRDL/**CRQL** reporting limit if either value is less than 5x CRDL/**CRQL** reporting limit.

7.10 The ICV is prepared from the ICV-6 standard **which is supplied by the EPA**. See the water or soil distillation SOPs for details on the preparation and distillation of this sample.

7.11 All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date three years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.

8.0 Sample Collection, Preservation, & Storage

8.1 Oxidizing agents destroy cyanide during storage and handling. Add ascorbic acid to prevent this.

8.2 Because of the reactivity of cyanide, analyze samples immediately after collection.

8.3 If samples must be held, add NaOH to bring the pH to 12. The color reaction is pH sensitive. Therefore, distillates or preserved water samples and standards should be carefully matched with respect to NaOH concentration.

8.4 Samples are collected, preserved, and stored according to Sample Control SOPs 4.1 "Receiving Samples" and 4.6 "Storing Samples."

8.5 Cyanide technical holding time is calculated from verified time of sampling. The technical holding time is 14 days.

9.0 Quality Control

9.1 After instrument calibration, the correlation coefficient must be determined to be 0.995 or better before sample analysis can begin. A forced intercept is not used for the calibration. An initial calibration verification (ICV) and initial calibration blank (ICB) must be analyzed. The ICV must be distilled with each batch of samples analyzed and the samples distilled with an ICV must be analyzed with that particular ICV. For aqueous cyanide samples, the ICV for cyanide also serves as the Laboratory Control Sample (LCS). The ICV must fall within 85-115% of the true value. The absolute ICB value must not be greater than the Contract Required Detection Limit (CRDL/**CRQL**) or reporting limit. If these control limits cannot be met, determine and correct the problem, recalibrate and reanalyze until all control limits can be met. If the ICV still does not pass, re-distillation of the samples and associated QC may be required. If instrument problems are suspect, notify your supervisor, document the problem in the maintenance log, and notify the instrumentation repair personnel.

9.2 A continuing calibration verification (CCV) and a continuing calibration blank (CCB) must be analyzed at a 10% frequency or every 2 hours throughout the analytical run. The control limits for these are the same as for the ICV and ICB above. In the event of a CCV/CCB failure, determine and correct the problem, recalibrate the instrument, and reanalyze all affected samples.

9.3 A method blank should be analyzed for every 20 samples of a similar matrix or for each case, whichever is more frequent. For SC DHEC the blank is prepared for every 10 water samples. The method blank value must not be greater than the reporting limit. If the blank exceeds the Contract Required Detection Limit (CRDL/**CRQL**) or reporting limit, analysis is stopped, the problem is determined and corrected, and any affected samples are reanalyzed, or re-prepped and reanalyzed. If the problem is with the instrumentation, the instrument should be fixed, recalibrated, and the affected samples rerun. Document the problem in the instrument maintenance log.

9.4 A laboratory control sample (LCS) must be analyzed with every 20 samples or with each case, whichever is more frequent. For SC DHEC the LCS is prepared for every 10 water samples. The LCS must agree within ± 20% of the true value for soils and 85-115% for waters. If the LCS falls outside these control limits, the analysis must be evaluated and it must be determined if an instrument problem exists. If so, the problem must be corrected and the affected samples reanalyzed. If not, the affected case must be reprepared and reanalyzed. As indicated, for aqueous cyanide samples, the ICV also serves as the LCS and it must be distilled. A separate LCS is required for soil cyanide samples. For non-CLP work, the soil ICV could be utilized as the LCS, but this is not a routine practice.

9.5 For every 20 samples of a similar matrix or for each Case/SDG, whichever is more frequent, a matrix spike analysis must be performed. For SC DHEC the duplicate frequency is 5% for water samples. The matrix spikes count towards this duplicate requirement. If the spike recovery is not within 75-125% recovery limits, and the sample result does not exceed 4x the spike added, a matrix interference is suspected. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x the CRDL/**CRQL** reporting limit, whichever is greater.

9.6 The distilled standards, i.e.**,** a mid range standard for CLP and MCAWW and low and high range standards for SW**-**846, are evaluated. The recoveries should be within +/-15% of the true value for CLP and MCAWW and +/-10% for **SW-846**. If this criteria are not met, the analyst should find the cause of the apparent error before proceeding. If re-analysis still fails acceptance criteria, the analysis must be re-started. If the standards continue to fail, re-distillation is required.

9.7 Contract Required Quantitation Limit (CRQL) Check Standard (CRI) – Used for EPA contract work. The concentration of the CRI is at the CRQL. The CRI is required at the beginning and end of each analytical sequence and every 20 samples. The acceptance range is 70-130%.

9.8 Contingency

9.8.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.8.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration & Standardization

10.**1** The instrument calibration curve is analyzed according to the sequence in section 11.0 (Procedure) below. The correlation coefficient must be 0.995 or greater for the calibration curve to be approved.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Timing

11.1.1 Sample throughput: 80 samples/h; 40s/samples

11.1.2 Pump speed: 35

11.1.3 Cycle period: 45s

11.1.4 Inject to start of peak period: 28s

11.1.5 Inject to end of peak period: 61s

11.2 System Notes

11.2.1 Allow 15 min for heating unit to warm up to 60°C.

11.2.2 Analyze distillates from water and soil/sediment preparation.

11.3 Operating System

11.3.1 Inspect the manifold for proper connections

11.3.2 Turn on the power to the instrument.

11.3.3 Secure the pump cassettes and place transmission lines in the corresponding solutions.

11.3.4 Select and download the method for cyanide (CN for **SW-846** or CLPCN for CLP.)

11.3.5 Allow the system to pump until the baseline is steady.

11.3.6 Place the standards in the rack in descending order of concentration.

11.3.7 “Submit and Calibrate Now” in software to begin calibration.

11.3.8 Under “Edit and Identification” enter the sample laboratory numbers and client ID numbers.

11.3.9 Once calibration is approved and sample tray is poured up, save the tray.

11.3.10 When the tray is complete, rinse the lines and place in DI water. The run log (**Attachment 2**) can now be printed for review and reporting.

11.3.11 When the manifold has been flushed with DI water, remove the lines and allow the system to continue pumping until all lines are dry.

11.3.12 Turn off pump and remove all cassettes.

11.3.13 Turn off power to the instrument.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\,R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked} \times 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \times 100$$

12.7 Concentration

12.7.1 Calculate the total or free cyanide in aqueous samples in µg/L as follows:

$$CN,\ \mu g/L = \frac{(A)(D)(F)}{(B)}$$

Where, A = µg/L CN of sample from regression analysis
B = Liter of original sample for distillation (0.050 L, typically)
D = Any dilution factor
F = Sample receiving solution volume (0.050 L, typical)

12.7.2 Calculate the total or free cyanide in soil/sediment samples in mg/kg as follows:

$$CN,\ \mu g/kg = \frac{(A)(D)(F)}{(B)(E)}$$

Where, A = µg/L CN of sample from regression analysis
B = Wet weight of original sample
D = Any dilution factor
E = % solids
F = Sample receiving solution volume

12.7.3 Calculate amenable cyanide as follows:

$$Amenable\ CN,\ \mu g/L = total\ CN - chlorinated\ CN$$

12.6 Example dilution calculation

Any sample with a value for total cyanide that exceeds the upper calibration range, must be analyzed at a dilution. For example, if a 5x dilution is determined to be needed, 1 **mL** sample is added to 4 **mL** 0.25 NaOH.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, NaOH, Zn Acetate, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW ILM04.1 **and** ILM05.3**,** plus revisions (335.2/335.3 CLP-M)

16.2 The New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.3 Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, 3rd Edition, Update III, 12/96, Method 9010B and 9012A

16.4 Lachat QuickChem AE, Method 10-204-001-1-A, November 1992

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, approved **June 2003**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, **April 2005**, plus revisions.

16.12 CompuChem Quality Manual, Revision **6**, **Update 1, 5/20/05**, plus revisions

16.13 Sample Control SOPs 4.1 "Receiving Samples" & 4.6 "Storing Samples"

16.14 Sample Preparation Procedure –072, "Aqueous Sample Midi-Distillation by CLP and NYSASP"

16.15 Sample Preparation Procedure –093, "Aqueous Sample Total and Free Cyanide Midi-Distillation by **SW-846** and NYSASP"

16.16 Sample Preparation Procedure –139, "Solid Sample for Total Cyanide Midi-Distillation by CLP and NYSASP"

16.17 Sample Preparation Procedure –191, "Solid Sample for Total and Free Cyanide Midi-Distillation by **SW-846** and NYSASP"

16.18 Sample Preparation Procedure –094, "Midi Distillation of Aqueous Samples for Amenable Cyanide by CLP and NYSASP"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – **Standards/QC Preparation for Cyanide by Lachat Log**

17.2 Attachment 2 – **Cyanide Analysis Log**

17.3 Attachment 3 – Cyanide Reporting Limits

Attachment 1

Standards/QC Preparation for Cyanide Analysis by Lachat

Seq. File Name:_____

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 O(5)

Page 1 of 3

REAGENTS*	REF#	Manufacturer	Lot#	Expiration Date
Sodium Hydroxide	1u(6)1-9-13			
Potassium Phosphate Monobasic Anhydrous (KH_2PO_4)	1U(6)1-8-2			
Chloramine-T	1U(6)1-9-3			
Barbituric Acid	1U(6)1-9-11			
Pyridine	1U(6)1-9-27			
Hydrochloric Acid	1M2-176-22			

REAGENT/STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
Carrier, 0.25M NaOH- 11 08 05 JM	In a 1 L volumetric flask, dissolve 10.0 g of NaOH in 800 mL of water. Dilute to the mark and invert three times. To prevent bubble formation, degas using a vacuum pump.
Phosphate Buffer- 09 29 05 JM	In a 1 L volumetric flask, dissolve 97 g of potassium phosphate monobasic anhydrous in 800 mL of water. Dilute to the mark and invert three times. To prevent bubble formation, degas using a vacuum pump.
Chloramine-T- 011 08 11 JM	To a 500 mL volumetric flask, add 250 mL of water followed by 2 g of □hloramines-T. Dilute to the mark and invert three times. To prevent bubble formation, degas using a vacuum pump. Prepare fresh daily.
Pyridine-Barbituric Acid- 10 26 05 JM	CAUTION – Always use a fume hood when preparing this reagent. Always wear a face shield when dispensing concentrated acids. This reagent has a six-month shelf – life if stored at 4 °C. In the fume hood, place 15.0 g of barbituric acid in a 1 L beaker and add 100 mL of water, rinsing down the sides of the beaker to wet the barbituric acid. Stir in 75 mL of pyridine (C_5H_5N) and mix until the barbituric acid dissolves. Add 15 mL of concentrated hydrochloric acid (12M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert three times. To prevent bubble formation, degas using a vacuum pump.

Reagent/Standard Solutions Prepared By: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log **Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

10/8/05:jad

Attachment 1 (Continued)

Standards/QC Preparation for Cyanide Analysis by Lachat

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 O(5) Page 2 of 3

STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
CN400– 10 26 05 JM (SW-846 only)	400 µg/L standard Place in a 100 mL volumetric flask: Pipet 4.0 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH and mix well
CN300–10 26 05 JM (SW-846 only)	300 µg/L standard Place in a 100 mL volumetric flask: Pipet 3.0 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution
CN200– 11 08 05 jm (SW-846 & CLP) USED FOR CCV FOR SW-846	200 µg/L standard Place in a 100 mL volumetric flask: Pipet 2.0 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution
CN100– 11 08 05 JM (SW-846 & CLP) USED FOR CCV FOR ILM05.3 & CLP	100 µg/L standard Place in a 100 mL volumetric flask: Pipet 1.0 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution
CN50– 11 08 05 jm (CLP only)	50 µg/L standard Place in a 100 mL volumetric flask: Pipet 0.5 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution.

Reagent/Standard Solutions Prepared By: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

**Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

10/8/05 jad

Attachment 1 (Continued)

Standards/QC Preparation for Cyanide Analysis by Lachat

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 O(5) Page 3 of 3

STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
CN40– 11 08 05 jm (SW-846 only)	40 µg/L standard Place in a 1000 mL volumetric flask: Pipet 0.4 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution.
CN20- 11 08 05jm (SW-846 & CLP)	20 µg/L standard Place in a 100 mL volumetric flask: Pipet 0.2 mL of 10 mg/L cyanide solution into the flask. Dilute to 100 mL with 0.25 N NaOH solution
CN10– 11 08 05 jm (SW-846 & CLP) (also used as CRI for CLP)	10 µg/L standard Place in a 1000 mL volumetric flask: Pipet 0.1 mL of 10 mg/L cyanide solution into flask. Dilute to 100 mL with 0.25 N NaOH solution
Blank– 11 08 05 jm (SW-846 & CLP) (Adjusted DI H_2O)	0.25 N NaOH solution Dissolve a 10 g of NaOH into a 1000 mL volumetric flask: with DI water

Reagent/Standard Solutions Prepared By: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

**Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

10/8/05:jad

Attachment 2

CYANIDE ANALYSIS LOG 1

CompuChem
A Division of Liberty Analytical Corp.

Method CLP - ILM04.1/ILM05.3 335.2/335.3/335.2(CLP-M)
SW-846 - 9010B/9012A

Analyst:_____

Date:_____

File Name(s):_____

Case Name(s):_____

Instrument ID: C2

Note: Manual Dilution Factor is not reflected in the sample results

Lachat Method 10-204-001-1-A

Cup Number	Sample ID	Sampling Date	Sampling Time	Manual Dil Factor	Result/Peak Area	Unit	Analyte Name	Comments
1	S200	8-Nov-05	13:34:57	1	6183484	uv-s	Cyanide CLP	
2	S100	8-Nov-05	13:35:44	1	3308347	uv-s	Cyanide CLP	
3	S50	8-Nov-05	13:36:30	1	1734952	uv-s	Cyanide CLP	
4	S20	8-Nov-05	13:37:16	1	783958	uv-s	Cyanide CLP	
5	S10	8-Nov-05	13:38:02	1	453565	uv-s	Cyanide CLP	
6	S0	8-Nov-05	13:38:49	1	121810	uv-s	Cyanide CLP	
13	ICV TV=99	8-Nov-05	13:41:08	1	107.060387	µg/L	Cyanide CLP	
14	ICB	8-Nov-05	13:41:54	1	-0.781976	µg/L	Cyanide CLP	
2	CCV TV=100	8-Nov-05	13:42:40	1	104.646996	µg/L	Cyanide CLP	
6	CCB	8-Nov-05	13:43:26	1	-1.537778	µg/L	Cyanide CLP	
1	MRS	8-Nov-05	13:45:46	1	102.958794	µg/L	Cyanide CLP	
2	85258 PBS	8-Nov-05	13:48:06	1	-2.214688	µg/L	Cyanide CLP	
3	85259 LCSS	8-Nov-05	13:48:52	1	201.235184	µg/L	Cyanide CLP	
4	828501 IS28SD20	8-Nov-05	13:49:38	1	-1.694837	µg/L	Cyanide CLP	
5	85261 IS28SD20S	8-Nov-05	13:50:23	1	91.497101	µg/L	Cyanide CLP	
6	85260 IS28SD20D	8-Nov-05	13:51:09	1	-3.642719	µg/L	Cyanide CLP	
7	828502 IS28SD16	8-Nov-05	13:51:53	1	-3.878506	µg/L	Cyanide CLP	
8	828503 IS28SD17	8-Nov-05	13:52:39	1	-4.05504	µg/L	Cyanide CLP	
9	828504 IS28SD18	8-Nov-05	13:53:24	1	-2.96871	µg/L	Cyanide CLP	
10	828505 IS28SD19	8-Nov-05	13:54:09	1	-3.634481	µg/L	Cyanide CLP	
2	CCV TV=100	8-Nov-05	13:54:55	1	109.702118	µg/L	Cyanide CLP	
6	CCB	8-Nov-05	13:55:41	1	-3.387886	µg/L	Cyanide CLP	

Reviewed by:_____ Date:_____

Attachment 3

Reporting Limits for Cyanide

Method	Soil mg/Kg	Water µg/L
ILM04.1/ILM05.3	**1.0**	**10**
335.2,335.3,9010B, 9012A, (335.2 (CLP-M)		**10**

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

ACCREDITED IN ACCORDANCE WITH

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-093/-81 SOP Section #: 3.4.2 Revision #: 8

SOP Title: Aqueous Sample Total & Free Cyanide Midi Distillation by SW-846 and NYSASP

Effective date: (QA fills in) 9-5-06

- Procedure prepared by: Martha Bell Date: 7-31-06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 9-5-06
- Reason for change: Addition of Chlorine check - Section 4.1
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition; Update III, Method 9010B, 9012A; MCAWW, March 1983, Methods 335.2, 335.3 NYSASP, June 2000, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure –093/-81: Aqueous Sample Total and Free Cyanide Midi Distillation by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure –093/-81: Aqueous Sample Total and Free Cyanide Midi Distillation by SW-846 and NYSASP

1.0 Scope and Application

This method is applicable to the determination of total cyanide in aqueous matrices including wastes or leachates and complies with requirements in SW-846 and the New York Analytical Services Protocol (NYSASP). Free cyanide procedures are included from Standard Methods.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The basis for the method includes a reflux-distillation of a known amount of sample releasing cyanide from its complexes in the form of hydrogen cyanide gas. The released gas is drawn by vacuum and absorbed in a sodium hydroxide solution. The cyanide is released from the sodium hydroxide solution by means of UV digestion and distillation and converted into cyanogen chloride by reaction with chloramine -T. In this form it reacts with pyridine and barbituric acid to give a red-colored complex. The intensity is measured colorimetrically at 578 ηm.

3.0 Definitions

3.1 Cyanide – cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system with mineral acid in the presence of magnesium ion.

3.2 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.3 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For some inorganic methods, the reporting limit is based on the MDL, and is usually 4 - 8 times higher than the MDL.

3.4 Reporting Units – µg/L

3.5 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for the DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.6 SC DHEC – South Carolina Department of Health and Environmental Control

3.7 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

Interferences are eliminated or reduced by distilling the samples. **Details of the procedures for chlorine and sulfide checks can be found in Sample Control SOP 4.13, "Handling & Verifying Proper Preservation of Samples Being Analyzed for Cyanides and Phenols".**

4.1 Chlorine Check for CLP

Oxidizing agents such as chlorine decompose most of the cyanides. Moisten a potassium iodide-starch test strip with acetate buffer. Mix then pour an aliquot of sample into a disposable cup. Test a drop of the sample with the moistened KI-starch paper. A blue color indicates the need for treatment. To treat, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

4.1.1 Chlorine Check Materials

- **Acetate Buffer: Dissolve 243 g of sodium acetate in 400 mL of DI water. Add 480 mL of glacial acetic acid in a 1000 mL flask.**
- **Potassium iodide-starch test strips**
- **Disposable 20 mL sample cups**
- **Ascorbic acid**

4.2 Sulfide Check for SW-846

Sulfide interference can be removed by adding an excess of bismuth nitrate to the waste (to precipitate the sulfide) before distillation. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation should be treated by the addition of bismuth nitrate. Samples are checked for sulfide upon receipt by the Receiving Department. See Sample Control SOP 4.13, "Handling & Verifying Proper Preservation of Samples Being Analyzed for Cyanides and Phenols."

4.**2**.1 Sulfide Check Materials for SW-846

Bismuth nitrate (0.062M), $Bi(NO)_3 \bullet 5H_2O$. Dissolve $Bi(NO)_3 \bullet 5H_2O$ in 100 mL of water. While stirring, add 250 mL of glacial acetic **acid**, CH_3COOH. Stir until dissolve and dilute to 1 liter with water.

4.**2**.2 Sulfamic acid (1N), H_2NSO_3H. Dissolve 100 g H_2NSO_3H in 1 liter of water.

4.**3** Nitrate/Nitrite Checks for SW-846

High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10 mg/L and in conjunction with certain organic compounds.

4.3.1 Nitrite check materials for SW-846

Microquant 1.14774.0001 nitrite kit - Use syringe to fill both glass bottles with 6 mL of aqueous sample. Add 1 flat microspoon of NO_2-AN to right hand bottle, dissolve and set aside for 3 minutes. Compare color with wheel. Treat with sulfamic acid if above 10 mg/L.

4.3.2 Nitrate check materials for SW-846

Spectroquant 1.14773.0001 nitrate kit – Add 1 mircospoon of NO_3-1A to tube. Add 5 mL of NO_3-2A to dissolve NO_3-1A. Add 1.5 mL of sample and mix immediately. Let stand 10 minutes. Measure at 515 ηm on Spect 20. Treat with sulfamic acid if above 10 mg/L.

4.**3**.**3** Sulfamic acid (1N), H_2NSO_3H. Dissolve 100 g H_2NSO_3H in 1 liter of water.

4.**4** pH Check

4.**4**.1 Samples received must be preserved with sodium hydroxide at the time of collection. Review the sample receiving report and check to see that the sample pH is equal to or greater than 12. If the pH of the sample is not within that range, contact Customer Service/Project Management. They will notify the client. The client will then say whether **or** not to proceed (must be in writing). If approval is granted, then add 2.0 mL of 10N sodium hydroxide per liter of sample to adjust the pH appropriately. Sample pH is checked upon receipt by the Receiving Department. See Sample Control SOP 4.3, "Checking and Recording pH of Metals, Cyanides, Phenols, and Wet Chemistry Samples."

4.**4**.2 pH check materials - pH paper colorHast strips

5.0 Safety

5.1 As with all laboratory procedures, you must wear the proper protective equipment (gloves, safety glasses, lab coat) when performing this procedure.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Midi reflux distillation apparatus

6.2 Heating block capable of maintaining 125 ± 5° C

6.3 Assorted volumetric glassware, pipets, or micropipets

7.0 Reagents and Standards

Note: All reagents and standards are documented in the Standards/QC Preparation for Cyanide by Lachat log book (Attachment 1).

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20^{th} Edition of Standard Methods, Method 1080), and is demonstrated to meet the blank criteria contained in this Standard Operating Procedure (SOP). It is referred throughout this SOP as DI water.

7.2 Sodium hydroxide solution, 0.25 N (for total cyanide only): Dissolve 10 g of NaOH in DI H_2O, then dilute to 1 liter.

7.3 Magnesium Chloride Solution (for total cyanide only): Weigh 510 grams of $MgCl_2/.6H_2O$ into a 1 L flask and dissolve it in DI water. Dilute to 1 liter with DI water. Shake the flask well to thoroughly mix.

7.4 Sulfuric Acid: 50% (v/v): Carefully add 100 mL of conc. Sulfuric acid to 100 mL DI H_2O. Adjust volumes as needed.

7.5 Stock cyanide solution, 1000 ppm: Dissolve 2.51 g of KCN and 2 g of KOH in a one liter volumetric flask. Standardize to determine the appropriate final volume. This standard can also be commercially purchased.

7.6 Stock intermediate cyanide solution, 10 ppm: Prepare daily. To a 250 mL volumetric flask, add about 125 mL of 0.25 N sodium hydroxide. Pipet calculated volume of standardized stock cyanide solution from equation on standards prep log into a class "A" 250 mL volumetric flask. Dilute to volume with 0.25 N NaOH. Mix well and transfer to a labeled polypropylene bottle and refrigerate at 4° C ± 2° C.

Note: Use this solution for sample spike LRS, HRS, and calibration standards.

7.7 ICV-6 cyanide solution supplied by EPA and must be ordered. It takes approximately 2 weeks to obtain.

7.8 Glacial acetic acid (1:9): Mix one volume of glacial acetic acid with 9 volumes of DI water.

7.9 Acetate buffer (for free cyanide only): Dissolve 410 g sodium acetate trihydrate in 500 mL DI water. Add glacial acetic acid (1:9) to yield a solution pH of 4.5 (approximately 500 mL).

7.10 Zinc acetate solution, 100 g/L (for free cyanide only): Using a 1 L volumetric flask, dissolve 120 g zinc acetate in 500 mL of DI water. Dilute to volume with DI water.

7.11 Methyl red indicator (for free cyanide only)

8.0 Sample Preservation and Storage

8.1 The sample should be collected in a 500 mL plastic bottle. All such bottles must be thoroughly cleaned and dried before collection.

8.2 Samples are preserved with 2 mL of 10N sodium hydroxide per liter of sample (pH >12) at the time of collection.

8.3 Samples must be stored in a refrigerator maintained at a temperature of 2 - 4.4° C.

8.4 Samples should be analyzed as rapidly as possible to meet holding time. Any redistillation due to problems during sample analysis must be taken into consideration to meet these holding times.

Note: Samples must be distilled and analyzed within 14 days from date of sampling for SW-846.

8.5 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 Preparation Blank

For every 20 samples or for each prep batch, whichever is more frequent, prepare a preparation blank. SC DHEC requires a preparation blank every 10 samples. A preparation blank is prepared by aliquoting 50 mL of DI water and distilling

according to sample preparation instructions. This blank is used to ascertain whether sample concentrations reflect contamination.

9.2 Sample Spike

For every 20 samples or for each SDG, whichever is more frequent, at least one spike sample preparation must be prepared. SC DHEC requires duplicates at a frequency of 10%. The sample spikes count towards this requirement. For sample spikes, pipet 0.5 mL of 10 ppm stock intermediate cyanide solution before distillation into the flask containing the sample to be spiked. True value of spike is 100 ppb.

9.3 Duplicate

For every 20 samples or for each SDG, whichever is more frequent, at least one duplicate sample must be prepared. SC DHEC requires duplicates at a frequency of 10%. The sample spikes count towards this requirement. Samples identified as field blanks should not be used for sample spike or duplicate analysis.

9.4 Initial Calibration Verification, ICV

One ICV must be distilled with each day of distillation.

To prepare, pipet 0.5 mL of ICV-6 cyanide solution into a distillation flask containing 50 mL of DI water. Distill according to the sample preparation instructions.

Note: This solution is supplied by EPA and must be ordered. Maintain a supply of this solution and store it in a refrigerator kept at 4° C ± 2° C. It takes about 2 weeks to obtain.

9.5 High-range standard (HRS)

At least one calibration standard (high-range) must be distilled with each analytical run to ensure that the distillation technique is reliable. The concentration of the HRS is 300 ppb. To prepare, pipet 1.5 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, that contains 50 mL of DI water. Distill according to sample preparation instructions.

9.6 Low-range standards (LRS)

At least one calibration standard (low-range) must be distilled with each analytical run to ensure that the distillation technique is reliable. The

concentration of the HRS is 40 ppb. To prepare, pipet 0.200 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, that contains 50 mL of DI water. Distill according to sample preparation instructions.

9.7 Laboratory Control Sample (LCS)

One liquid LCS must be prepared for every 20 samples or prep batch, whichever is more frequent. For SC DHEC the LCS is prepared every 10 samples. This liquid LCS is 0.5 mL of ICV 6 solution into 50 mL DI H_2O and must be ordered. Maintain a supply of this material and store in refrigerator at 4° C ± 2° C. It takes about 2 weeks to obtain.

9.8 Contingency

9.8.1 If due to a lab accident or to QC failures a re-preparation and re-analysis are required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for directions on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analysis may proceed.

9.8.3 Refer to Data Review SOP 14.3.2.1 "Data Management: Wet Chemistry Data Review and Verification" for information on how to handle reporting of data that are unacceptable or out-of-control.

9.8.4 Any other issues that potentially affect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

Refer to SOPs 9.1, "Calibrating Automatic Pipettes in the Inorganic Laboratory and 13.17, "Analytical Balance Calibration and Maintenance".

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 Manual distillation: set up the apparatus as shown in Attachment 2.

Note: If samples are known or suspected to contain sulfide, add 5.0 mL of 0.062M bismuth nitrate solution through the air inlet tube. Mix for three minutes. Use lead acetate paper to check the sample for the presence of sulfide. A positive test is indicated by a black color on the paper.

If samples are known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 1.0 mL of 1N sulfamic acid solution through the air inlet tube. Mix for three minutes.

11.2 Total Cyanide Distillation

Thoroughly mix sample before aliquotting. Transfer 50 mL of aqueous sample into the distillation flask.

11.2.1 Add 50 mL of 0.25N NaOH solution to the absorber tube.

11.2.2 Connect the apparatus as shown in Attachment 2.

11.2.3 Adjust suction so that about 3 air bubbles/sec enters through the distillation flask. This air will carry HCN gas from the flask to the absorber tube for collection.

11.2.4 After approximately 5 minutes of vacuum flow, add 5 mL of 1:1 H_2SO_4 through the inlet tube. This volume should be sufficient to bring the pH of the solution to below 2. After the addition of acid, rinse the inlet tube with a small amount of DI water.

Warning: Violent reaction may occur with the acid addition. Add slowly and observe the sample's response.

11.2.5 Allow air to mix the contents of the distillation flask for approximately 5 minutes.

11.2.6 Add 2 mL $MgCl_2$ reagent through the inlet tube. Excessive foaming from sample containing surfactants may be quelled by the addition of another 2 mL $MgCl_2$. Rinse the tube with a small amount of DI water.

11.2.7 Turn on the heating block and set for 123 - 125° C. Heat the flask to rapid boiling. Do not allow the solution to back up and overflow into the air inlet tube.

11.2.8 Distill for an hour and a half and then discontinue heating, but continue air flow.

11.2.9 Cool and aerate for 15 minutes. Disconnect absorber tube and close off the vacuum source.

11.2.10 Carefully transfer the solution from the scrubber to a 50 mL plastic centrifuge tube that is properly labeled with CompuChem number and "CN-" identification to ensure that the cyanide sample is not confused with a sample that was prepared for metals. Replace cap.

11.2.11 Record preparation information on the Cyanide Preparation Log worksheet (Attachment 3). All information must be properly entered, the sheet reviewed, signed and copied. Samples are now ready for analysis by IP 930, "Total and Free Cyanide Analysis of Water and Soil/Sediment Distillates by CLP, MCAWW, SW-846, NYSASP & Lachat."

11.3 Free Cyanide Distillation

11.3.1 Follow steps 11.2.1 through 11.2.3.

11.3.2 After approximately 5 minutes of vacuum flow, add 20 mL of acetate buffer and 20 mL of zinc acetate solution through the inlet tube. Also add 2 or 3 drops of methyl red indicator. Rinse the tube with a small amount of DI water.

11.3.3 Allow air to mix the contents of the distillation flask for approximately 5 minutes. If solution is not pink, add acetic acid (1:9) drop wise through the inlet tube until a pink color persists.

11.3.4 Follow steps 11.2.7 through 11.2.11. Record preparation information on the Cyanide Preparation Log worksheet (Attachment 3).

12.0 Data Analysis & Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste. Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 SW-846, 3rd Edition, Update III, 12/96, Methods 9012A (with the exception of midi distillation) and 9010B (with the exception of midi distillation and cyanide content determination by 9012A).

16.2 The New York Analytical Services Protocol (NYSASP), June 2000, plus revisions

16.3 Methods for Chemical Analysis of Water and Wastes, 3/83, Methods 335.2 (off-line distillation) and 335.3 (colorimetric determination)

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, July 2003, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, December 2005, plus revisions.

16.11 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080 and 4500-CN I (free cyanide)

16.12 CompuChem Quality Manual, Revision 7, Update 1, 12/13/05, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Sample Control SOP 4.13, "Handling & Verifying Proper Preservation of Samples Being Analyzed for Cyanides and Phenols"

16.16 Sample Control SOP 4.3, "Checking and Recording pH of Metals, Cyanides, Phenols, and Wet Chemistry Samples"

16.17 IP 930, "Total and Free Cyanide Analysis of Water and Soil/Sediment Distillates by CLP, MCAWW, SW-846, NYSASP & Lachat."

16.18 Data Review SOP 14.3.2.1, "Data Management: Wet Chemistry Data Review and Verification"

16.19 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – QC Standards Log

17.2 Attachment 2 – Cyanide Distillation Apparatus Diagram

17.3 Attachment 3 – Cyanide Preparation Log

Attachment 1

Standards/QC Preparation for Cyanide Digestion

Case/SDG : _____

COMPUCHEM a division of Liberty Analytical. Corp. WORKSHEET 1 J (8)

Page 1 of 2

STANDARDS	REF#[1]	MANUFACTURER	LOT #	EXPIRATION DATE
CN 1000 mg/L	1U(6)1-11-15	NSI	022006	02/07
ICV-6 (0400)	1U (6)1-11-13	EPA	0400	3/09
EPA LCS-CN (0899)	1U (6)1-11-12	EPA	0899	03/09
REAGENTS				
Magnesium Chloride (MgCl)	1m2-178-22	Caledon	42526	03/09
Sodium Hydroxide (NaOH)	1U(6)1-9-13	JT Baker	A06932	04/06
Sulfuric Acid (H_2SO_4)	1m2-179-08	Caledon	58463	5/09
Bismuth Nitrate ($Bi(NO_3)$)	1U(6)1-11-24	EM Science	33236425	6/09
Sulfamic Acid (H_2NSO_3H)	1U(6)1-11-21	EM Science	40334520	5/09

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS
NaOH 07/25/06 – ML 0.25 N	Dissolve 20g of NaOH in DI H_2O, then dilute to 2000 mL
MgCl 07/24/06 – ML Magnesium Chloride	Weigh 510 grams of $MgCl_2 \cdot 6H_2O$ into a 1000 mL flask and dissolve it in DI water. Dilute to 1000 mL with DI water. Shake the flask well to thoroughly mix.
H_2SO_4 07/24/06 – ML 50% (v/v)	Carefully add 250 mL of conc. Sulfuric acid to 250 mL DI H_2O.
$Bi(NO)_3$ 06/23/06 – JM	Dissolve $Bi(NO)_3 \cdot 5H_2O$ in 100 mL of DI H_2O. Mix well before use.
H_2NHSO_3 06/23/06 – JM	Dissolve 100 g H_2NSO_3H in 1 L of DI H_2O. Mix well before use.

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Ref#[1] = Logbook ID, Page number, and item number from Materials Receipt Log

7/18/06:jad

Standard Lot Number[2] consists of standard ID, date prepared, and initials of preparer.

Attachment 1 (continued)

Standards/QC Preparation for Cyanide Digestion

COMPUCHEM a division of Liberty Analytical. Corp.. WORKSHEET 1 J (8) Page 2 of 2

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS	Resultant Analyte Conc. (µg/L)
Stock Intermediate CN Solution- 07/25/06 – ML	Place in a 50 mL volumetric flask: Pipet 0.5 mL of stock cyanide standard into the flask. Dilute to the mark with 0.25 NaOH solution and mix well. Prepare daily.	CN: 10, 000
ICV-6 07/25/06 – ML	Pipet 0.5 mL of ICV-6 (0400) cyanide solution into a distillation flask containing 50 mL of DI water. Distill according to the sample preparation instructions.	CN: 99
LCSW 07/25/06 – ML	Pipet 0.5 mL of ICV-6 (0400) cyanide solution into a distillation flask containing 50 mL of DI water. Distill according to the sample preparation instructions.	CN: 99
LCSS 07/25/06 – ML	Weigh 1.0 g of the EPA LCS-CN (0899) into a distillation flask containing 50 mL DI H_2O. Distill according to the sample preparation instructions.	CN: 192
MRS 07/25/06 – ML (for CLP)	Pipet 0.5 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 100
HRS 07/25/06 – ML (for SW-846)	Pipet 1.5 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 300
LRS 07/25/06 – ML (for SW-846)	Pipet 0.200 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 40

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Standard Lot Number[2] consists of standard ID, date prepared, and initials of preparer.

7/18/06 jad

Attachment 2

Cyanide Preparation CompuChem a division of Liberty Analytical Corp. SPP-072/-093/-094/-139/-191

Method (Circle One): ILM04.1 335.2 CLP-M 335.3 9012A (Mod) 4500-CN I ILM05.3 **Batch No.** _____ Worksheet: CN Prep

Case/SDG:_____ **Prepared by:**_____ **Date:**_____ **Matrix:** _____

#	CCN (Lab ID)	Client ID	Date Rec'd	Initial mL g	Final (mL)	Description Before	Description After	S-*	Cl_2*	pH	Amen Prep**	Free Prep**
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21	Matrix Spike:					Ref CCN:						
22	Matrix Spike Dup:					Ref CCN:						
23	Duplicate Sample:					Ref CCN:						
24	Lab Control Sample:											
25	Prep Blank:					Reviewed by:_____						
26	ICV-6 :					Date:_____						
27	CLP Mid-Range Std :					Ref #:						
28	SW-846 Low-Range Std :					Ref #:						
29	SW-846 High-Range Std :					Ref # :						

	Hot Block Temperature	Distillation Time	Chlorination Time
	Start:	Start:	Start:
	End:	End:	End:

ICV-6 Ref:
CN:
Ref #:

Matrix Spike (MS/MSD) Ref:
CN:
Ref #:

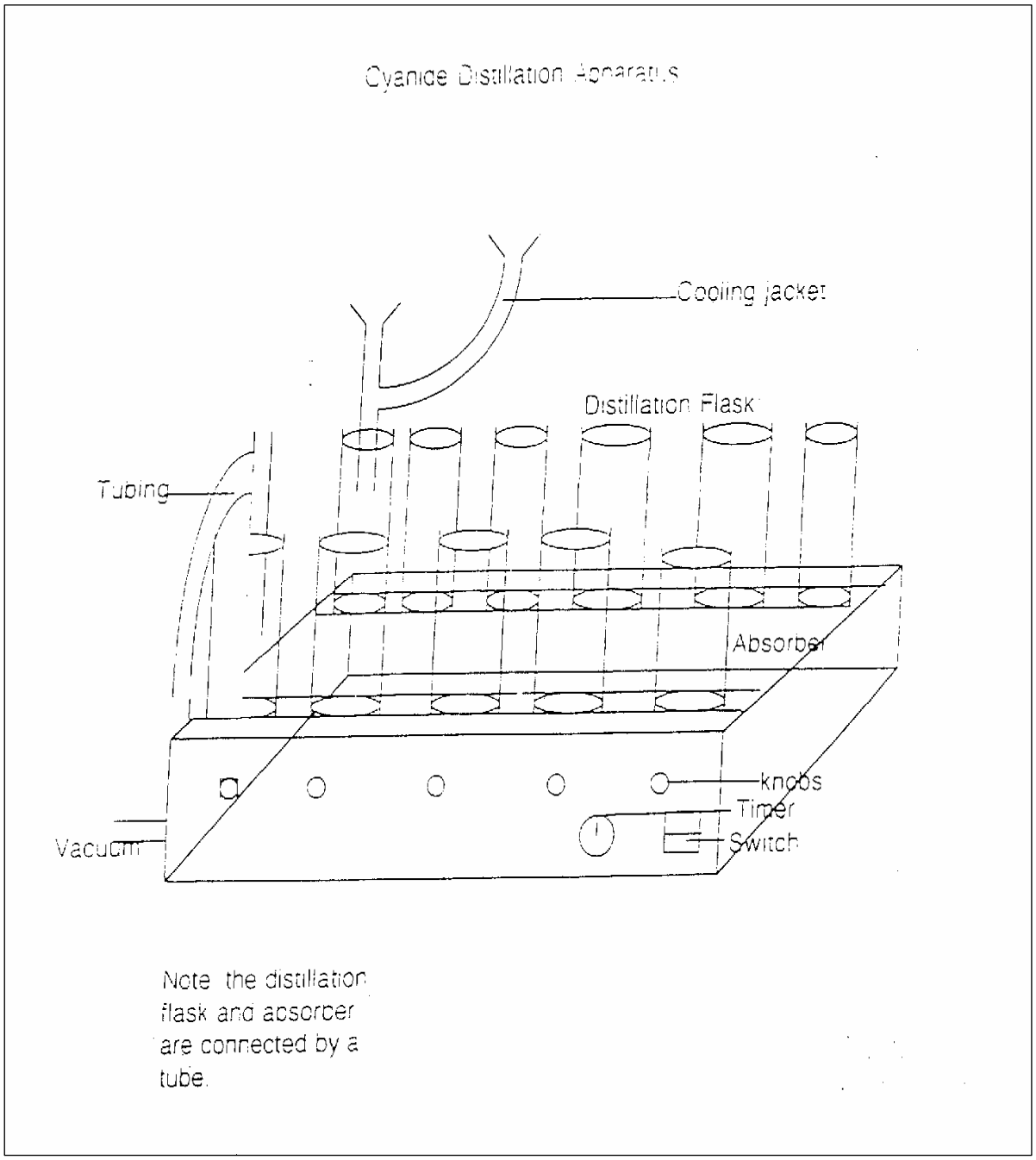
LCS Ref:
CN:
Ref #:

Key
* D= Detected ND= Not Detected
** Y= Yes N= No

"The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation."

1/11/06:jad

Attachment 3



CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-072 SOP Section #: 3.4.1 Revision #: 11

SOP Title: Aqueous Sample Cyanide Midi Distillation by CLP, NYSASP, and MCAWW

Effective date: (QA fills in) 11/17/05

- Procedure prepared by: Martha Scott — Date: 11-17-05
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) — Date: 11-17-05
- Reason for change: annual review and response to EPA audit.
- This procedure meets the requirements of the following approved method references: US EPA CLP SOWs ILM04.1 and ILM05.3, 335.2/335.3 (CLP-M); SW-846, 3rd Edition, Update III, Method 9010B, Method 9012A; MCAWW, March 1983 Method 335.2 Method 335.3

ORIGINAL

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -072: Aqueous Sample Cyanide Midi Distillation by CLP, NYSASP, and MCAWW

Table of Contents

Sample Preparation Procedure -072: Aqueous Sample Cyanide Midi Distillation by CLP, NYSASP, and MCAWW

1.0 Scope and Application

This method is applicable to the determination of total cyanide in aqueous matrices including wastes or leachates and complies with requirements in CLP SOW ILM04.1, ILM05.3, 335.2/335.3 (CLP-M), and the New York Analytical Services Protocol (NYSASP).

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The basis for the method includes a reflux-distillation of a known amount of sample releasing cyanide from its complexes in the form of hydrogen cyanide gas. The released gas is drawn by vacuum and absorbed in a sodium hydroxide solution. The cyanide is released from the sodium hydroxide solution by means of UV digestion and distillation and converted into cyanogen chloride by reaction with chloramine -T. In this form it reacts with pyridine and barbituric acid to give a red-colored complex. The intensity is measured colorimetrically at 578 **η**m.

3.0 Definitions

3.1 Cyanide – cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system with mineral acid in the presence of magnesium ion.

3.2 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration.

3.4 Reporting Units – μg/L

4.0 Interferences

Interferences are eliminated or reduced by distilling the samples. Details of the procedures for chlorine and sulfide checks can be found in Sample Control SOP 4.13, "Handling & Verifying Proper Preservation of Samples Being Analyzed for Cyanides and Phenols".

4.1 Chlorine Check for CLP

Oxidizing agents such as chlorine decompose most of the cyanides. Moisten a potassium iodide-starch test strip with acetate buffer. Mix then pour an aliquot of sample into a disposable cup. Test a drop of the sample with the moistened KI-starch paper. A blue color indicates the need for treatment. To treat, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

4.1.1 Chlorine Check Materials

- Acetate Buffer: Dissolve 243 g of sodium acetate in 400 **mL** of DI water. Add 480 g of glacial acetic acid in a 1000 **mL** flask.
- Potassium iodide-starch test strips
- Disposable 20 **mL** sample cups
- Ascorbic acid

4.2 Sulfide Check for CLP

Sulfides adversely affect the colorimetric and titration procedures. If a drop of the sample on lead acetate test paper indicates the presence of sulfides, treat 25 mL more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to

minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide.

4.2.1 Sulfide Check Materials

- Acetate Buffer: see chlorine check (paragraph 4.1.1)
- Lead acetate test strips
- Disposable 20 **mL** sample cups

4.3 pH Check

Details of the procedures for pH checks can be found in Sample Control SOP 4.3, "Checking and Recording pH of Metals, Cyanides, Phenols and Wet Chemistry Samples".

Samples received must be preserved with sodium hydroxide at the time of collection. Review the sample receiving report and check to see that the sample pH is equal to or greater than 12. If the pH of the sample is not within that range, contact Customer Service/Project Management. They will notify the client. The client will then say whether of not to proceed (must be in writing). If approval is granted, then add 2.0 **mL** of 10N sodium hydroxide per liter of sample to adjust the pH appropriately.

pH check materials: pH paper colorpHast strips

5.0 Safety

5.1 As with all laboratory procedures, you must wear the proper protective equipment (gloves, safety glasses, lab coat) when performing this procedure.

5.2 Laboratory staff is encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Midi reflux distillation apparatus

6.2 Heating block capable of maintaining 125 ± 5°C

6.3 Assorted volumetric glassware, pipets, or micropipets

7.0 Reagents and Standards

Remember to record all reagents and standards in the **Standards/QC Preparation** Log (**Attachment 1**).

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20^{th} Edition of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Sodium hydroxide solution, 0.25 N: Dissolve 10g of NaOH in DI H_2O, then dilute to 1 liter.

7.3 Magnesium Chloride Solution: Weigh 510 grams of $MgCl_2/.6H_2O$ into a 1 L flask and dissolve it in DI water. Dilute to 1 liter with DI water. Shake the flask well to thoroughly mix.

7.4 Sulfuric Acid: 50% (v/v): Carefully add 100 mL of conc. Sulfuric acid to 100 mL DI H_2O. Adjust volumes as needed.

7.5 Stock **standard** solution, 1000 ppm**, is purchased commercially.**

7.6 Stock intermediate cyanide solution, 10 ppm: **Place in** a 50 **mL** volumetric flask **approximately 25mL of 0.25N sodium hydroxide.** Pipet **0.5 mL of stock cyanide standard** into **the** flask. Dilute to **mark** with 0.25 NaOH **solution and mix** well**. Prepare daily.**

Note: Use this solution for sample spike (MS/MSD), MRS and calibration standards.

7.7 ICV-6 cyanide solution **is** supplied by **the** EPA. The ICV-6 solution is also used for the LCS.

8.0 Sample Collection, Preservation, and Storage

8.1 The sample should be collected in a 500 mL plastic bottle. All such bottles must be thoroughly cleaned and dried before collection.

8.2 Samples are preserved with 2 mL of 10N sodium hydroxide per liter of sample (pH >12) at the time of collection.

8.3 Samples must be stored in a refrigerator maintained at a temperature of 4°C ± 2°C.

8.4 Samples should be analyzed as rapidly as possible to meet holding time. Any redistillation due to problems during sample analysis must be taken into consideration to meet these holding times.

Note: Samples must be distilled and analyzed within 12 days from time of receipt in order to meet holding times for CLP.

8.5 The sample preparation technician should visually inspect all glassware before use. Any glassware with residues, dust, or discoloration of any kind should be rejected and recleaned.

8.6 Soap and hot water wash all glassware immediately after use. Rinse well with DI water.

9.0 Quality Control

9.1 Preparation Blank

For every 20 samples or for each prep batch, whichever is more frequent, prepare a preparation blank. A preparation blank is prepared by aliquoting 50 **mL** of DI water and distilling according to sample preparation instructions. This blank is used to ascertain whether sample concentrations reflect contamination.

9.2 **Matrix** Spike

For every 20 samples or for each SDG, whichever is more frequent, one **matrix** spike **sample** must be prepared. For **the matrix spike** sample**,** pipet 0.5 **mL** of 10 ppm stock intermediate cyanide solution **into a 50 mL aliquot of sample designated as the original, and distilled according to** sample **preparation instructions. The true** value of **the matrix** spike is 100 ppb.

9.3 Duplicate **Sample**

The duplicate sample is prepared by taking a 50 mL aliquot of the chosen QC sample. Samples identified as field blanks should not be used for **matrix** spike or duplicate **sample** analysis.

9.4 Initial Calibration Verification, ICV

One ICV must be distilled with each day of distillation.

To prepare **the ICV**, pipet 0.5 **mL** of ICV-6 cyanide solution into a distillation flask containing 50 **mL** of DI water. Distill according to the sample preparation instructions. The preparation of the ICV must be noted on the preparation log.

Note: The ICV-6 solution is supplied by **the** EPA.

9.5 Mid-range standards (MRS) (for CLP)

At least one calibration standard (mid-range) must be distilled with each analytical run to ensure that the distillation technique is reliable. The concentration of the MRS is 100 ppb. To prepare, pipet 0.5 **mL** of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 **mL** of DI water. Distill according to sample preparation instructions.

9.6 LCS

One liquid LCS must be prepared for every 20 samples or prep batch, whichever is more frequent. This liquid LCS is 0.5 mL of ICV-6 solution into 50 mL DI H_2O**.**

10.0 Calibration and Standardization

NA

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Manual distillation: set up the apparatus as shown in Attachment **2**.

Note: If samples are known or suspected to contain sulfide, add 5.0 mL of 0.062M bismuth nitrate solution through the air inlet tube. Mix for three

minutes. Use lead acetate paper to check the sample for the presence of sulfide. A positive test is indicated by a black color on the paper.

If samples are known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 5.0 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

11.2 Thoroughly mix sample before aliquotting. Transfer 50 mL of aqueous sample into the distillation flask.

11.2.1 Add 50 **mL** of 0.25 N NaOH solution to the absorber tube.

11.2.2 Connect the apparatus as shown in Attachment **2**.

11.2.3 Adjust suction so that about 3 air bubbles/sec enters through the distillation flask. This air will carry HCN gas from the flask to the absorber tube for collection.

11.2.4 After approximately 5 minutes of vacuum flow, add 5 **mL** of 1:1 H_2SO_4 through the inlet tube. Rinse the inlet tube with the addition of 1 to 2 **mL** of DI water. This volume should be sufficient to bring the pH of the solution to below 2.

Warning: Violent reaction may occur with the acid addition. Add slowly and observe the sample's response.

11.2.5 Allow air to mix the contents of the distillation flask for 5 minutes.

11.2.6 Add 2 **mL** $MgCl_2$ reagent through the inlet tube. Rinse the inlet tube with the addition of 1 to 2 **mL** of DI water. Excessive foaming from sample containing surfactants may be quelled by the addition of another 2 **mL** $MgCl_2$.

11.2.7 Turn on the heating block and set for 123-125°C. Heat the flask to rapid boiling. Do not allow the solution to back up and overflow into the air inlet tube.

11.2.8 Distill for an hour and a half and then discontinue heating, but continue air flow.

11.2.9 Cool and aerate for 15 minutes. Disconnect absorber tube and close off the vacuum source.

11.2.10 Carefully transfer the solution from the scrubber to a 50 mL plastic centrifuge tube which is properly labelled with CompuChem number and "CN-" identification to ensure that the cyanide sample is not confused with a sample that was prepared for metals. Replace cap.

11.2.11 Record prep information in the cyanide prep log. (Attachment **3**) All information must be properly entered, signed and reviewed.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW ILM04.1, ILM05.3, plus revisions (335.2/335.3 CLP-M)

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 The New York Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, June **2003**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, **April 2005**, plus revisions.

16.12 CompuChem Quality Manual, Revision **6**, **Update 1, 5/20/05**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Sample Control SOP 4.13, "Handling & Verifying Proper Preservation of Samples Being Analyzed for Cyanides and Phenols".

16.16 Sample Control SOP 4.3, "Checking and Recording pH of Metals, Cyanides, Phenols and Wet Chemistry Samples"

16.17 Method of Chemical Analysis of Water and Wastewater, March 1983, Methods 335.2 and 335.3

17.0 Attachments

Attachment 1 **– Standards/QC Preparation Log for Cyanide Digestion**

Attachment 2 - **Cyanide Distillation Apparatus Diagram**

Attachment 3 **– Cyanide Sample Preparation Log**

Attachment 1

Standards/QC Preparation for Cyanide Digestion

Case/SDG : _____

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 J (8)

Page 1 of 2

STANDARDS	REF#[1]	MANUFACTURER	LOT #	EXPIRATION DATE
CN 1000 mg/L	1U(6)1-10-26	NSI	040105	04/06
ICV-6 (0400)	1U (6)1-10-28	EPA	0400	11/08
EPA LCS-CN (0899)	7M1-14-7	EPA	0899	08/07
REAGENTS				
Magnesium Chloride (MgCl)	1U(6)1-9-14	JT Baker	V35334	04/06
Sodium Hydroxide (NaOH)	1U(6)1-9-13	JT Baker	Y04H05	04/06
Sulfuric Acid (H_2SO_4)	1m2-177-18	Caledon	53821	10/06

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS
NaOH - __ __ __ __ __ __ - __ __ 0.25 N	Dissolve 20g of NaOH in DI H_2O, then dilute to 2000 mL
MgCl - __ __ __ __ __ __ - __ __ Magnesium Chloride	Weigh 510 grams of $MgCl_2/.6H_2O$ into a 1000 mL flask and dissolve it in DI water. Dilute to 1000 mL with DI water. Shake the flask well to thoroughly mix.
H_2SO_4- __ __ __ __ __ __ - __ __ 50% (v/v)	Carefully add 250 mL of conc. Sulfuric acid to 250 mL DI H_2O.

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Ref#[1] = Logbook ID, Page number, and item number from Materials Receipt Log

Standard Lot Number[2] consists of standard ID, date prepared, and initials of preparer.

11/11/05 jtad

Attachment **1 (Continued)**

Standards/QC Preparation for Cyanide Digestion

COMPUCHEM a division of Liberty Analytical. Corp.. WORKSHEET 1 J (8)

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS	Resultant Analyte Conc. (µg/L)
Stock Intermediate CN Soln- __ __ __ __ __ __ - __ __	Place in a 50 mL volumetric flask: Pipet 0.5 mL of stock cyanide standard into the flask. Dilute to the mark with 0.25 NaOH solution and mix well. Prepare daily.	CN: 10, 000
ICV-6-__ __ __ __ __ __ - __ __	Pipet 0.5 mL of ICV-6 (0400) cyanide solution into a distillation flask containing 50 mL of DI water. Distill according to the sample preparation instructions.	CN: 99
LCSW - __ __ __ __ __ __ - __ __	Pipet 0.5 mL of ICV-6 (0400) cyanide solution into a distillation flask containing 50 mL of DI water. Distill according to the sample preparation instructions.	CN: 99
LCSS-__ __ __ __ __ __ - __ __	Weigh 1.0 g of the EPA LCS-CN (0899) into a distillation flask containing 50 mL DI H_2O. Distill according to the sample preparation instructions.	CN: 192
MRS - __ __ __ __ __ __ - __ __ (for CLP)	Pipet 0.5 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 100
HRS-__ __ __ __ __ __ - __ __ (for SW-846)	Pipet 1.5 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 300
LRS - __ __ __ __ __ __ - __ __ (for SW-846)	Pipet 0.200 mL of the 10 ppm stock intermediate cyanide solution into the distillation flask, which contains 50 mL of DI water. Distill according to sample preparation instructions.	CN: 40

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

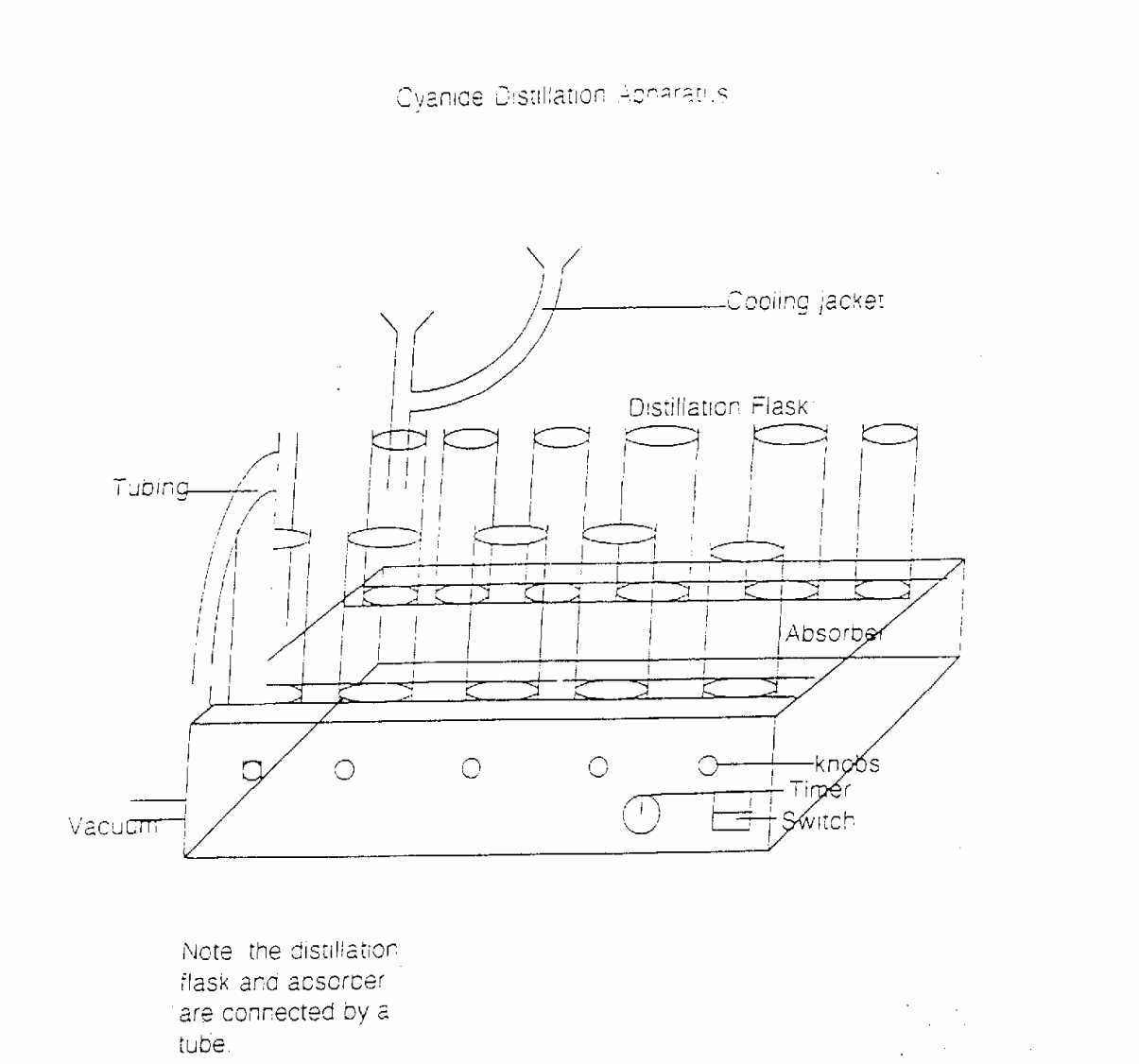
The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Standard Lot Number[2] consists of standard ID, date prepared, and initials of preparer.

11/11/05:jad

Attachment subject to change without notice.

Attachment 2



NOTE: Attachment is subject to change without notice.

Attachment **3**

Cyanide Preparation **Page 1 of 1** SPP -072 / -093 / -094 / -139 / -191

CompuChem a division of Liberty Analytical Corp. Method: CLP or 335.2 CLP-M or 335.3 or 9012A (Mod) or 4500-CN _____ (Enter One)

Batch No. _____ Date: 11/10/2005 Matrix: _____

Prepared By: _____ EPA Case/SDG: _____

#	CCN (Lab ID)	Client ID	Sample Type	Initial mL g	Final (mL)	Description Before	Description After	S-*	Cl2*	pH	Amen Prep**	Free Prep**
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												

Check if standard(s) distilled		Hot Block Temp °C	Chlorination Time	Distillation Time
ICV		start:	Start:	Start:
LRS 40 ppb=0.2ml 10ppm (SW846)		end:	Stop:	Stop:
MRS 100 ppb=0.5ml 10ppm (CLP & amenable)				
HRS 300 ppb=1.5ml 10ppm (SW846)				

*D = Detected / ND = Not Detected

** Y = Yes / N = No

10/13/05:jad

Comments: _____

Reviewed By: _____ Date: _____

NOTE: Attachment is subject to change without notice.

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

ACCREDITED IN ACCORDANCE WITH
NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) _____

- Procedure Code: IP 309 SOP Section #: 3.2.1.6 Revision #: 15

SOP Title: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 and NYSASP

Effective date: (QA fills in) 8/4/06

- Procedure prepared by: [signature] Date: 8/4/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 08.04.06
- Reason for change: CHANGED LOG BOOK CALIBRATION SEQUENCE.
- This procedure meets the requirements of the following approved method references: SW-846 3rd Edition, Update III, Method 6010B; New York State Analytical Services Protocol (NYSASP), June 2000, plus Rev

ORIGINAL

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 309: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 and NYSASP

Table of Contents

Instrument Procedure 309: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 and NYSASP

1.0 Scope and Application

Inductively coupled plasma/atomic emission spectroscopy (ICP/AES) is used in the determination of elements in solution including metals. The method is applicable to a large number of metals and wastes. All matrices, including groundwater, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, TCLP leachates, and other solid wastes, require digestion prior to analysis.

Method 6010B is applicable to the elements listed in Attachment 1 for Trace ICP P3 and Attachment 2 for Trace ICP P4. For TCLP the following elements are to be determined: arsenic, barium, cadmium, chromium, lead, selenium, and silver. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. The data shown in these attachments are provided from clean aqueous samples.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2.0 Summary of Method

Prior to analysis, samples must be solubilized or digested following Sample Preparation Procedure –1080, "Digestion Block Preparation of Aqueous Samples for the ICP Determination of Total or Dissolved Metals by SW-846, MCAWW, Standard Methods, and NYSASP" or Sample Preparation Procedure –240, "Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 and NYSASP." TCLP samples must be leached following Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)" after which the leachates are digested as liquid samples using the TCLP spiking levels.

Method 6010B is executed by the laboratory using the simultaneous, multielemental determination of elements by ICP. The method measures element-emitted light by

optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made. Three exposures are averaged to obtain a final result.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For some inorganic methods, the reporting limit is based on the MDL, and is usually 4-8 times higher than the MDL. This is referred to as the Practical Quantitation Limit (PQL).

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

For CLP the reporting limit is the Contract Required Detection Limit (CRDL) for inorganics.

3.3 Reporting Units – µg/L for water and mg/Kg for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM and SC DHEC soils while a rate of 10% is require for SC DHEC waters. If samples are batched together from different sites, project-specific QC must be processed.

3.5 Toxicity characteristics constituents and regulatory levels for TCLP

Constituent (mg/L)	**Chronic toxicity reference level (mg/L)**	**Regulatory level (mg/L)**
Arsenic	0.05	5.0
Barium	1.0	100.0
Cadmium	0.01	1.0
Chromium	0.05	5.0
Lead	0.05	5.0
Mercury	0.002	0.2
Selenium	0.01	1.0
Silver	0.05	5.0

3.6 SC DHEC – South Carolina Department of Health and Environmental Control

3.7 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Spectral Interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computerized correction of the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Users of simultaneous multi-element instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in the table in Attachment **1**. The data are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

4.1.1 The interference is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of determined As (at 193,696 ηm) in a sample containing approximately 10 mg/L of Al. According to Attachment **1**, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of A1 would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different effects and must be evaluated individually since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

4.1.2 The dashes in Attachment **1** indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.1.3 At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 393.231 ηm wavelength interferes with the listed potassium line at 766.491 ηm.

4.2 Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump or by using the standard additions method. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon using a tip washer prior to nebulization or by diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

4.3 Chemical Interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 The Thermo Jarrell Ash ICAP Trace Analyzer uses yttrium as an internal standard and takes into account any interference by ratioing the elements detected against the yttrium detected. All standards, blanks, and samples are spiked with yttrium.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Inductively coupled argon plasma emission spectrometer

6.1.1 Computer-controlled emission spectrometer with background correction

6.1.2 Radio frequency generator

6.1.3 Argon gas supply: Welding grade or better

6.2 Operating conditions

6.2.1 The analyst should follow the instructions provided by the instrument manufacturer.

6.2.2 For operation with organic solvents, use of the auxiliary argon inlet, solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power (to obtain stable operation and

precise dynamic range) are recommended. Interference effects must be established for each individual analyte line on that particular instrument.

6.2.3 All measurements must be within instrument linear range where coordination factors are valid. The analyst must

- verify that the instrument configuration and operating conditions satisfy the analytical requirements
- maintain quality control data confirming instrument performance and analytical results

7.0 Reagents & Standards

Refer to the Standards/QC Preparation for Trace ICP logbook (Attachment 4) for details of all standard preparations. Label each standard bottle with the lot number as described in the logbook.

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20^{th} Edition of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Concentrated acids – ultra high-purity grade

7.2.1 Hydrochloric acid (HCl), Caledon, Trace Metal Grade

7.2.2 Nitric acid (HNO_3), J.T. Baker, Instra-Analyzed® Reagent

7.2.3 The same grade/purity of acids is to be used for all sample preparation, calibration standards, blanks, and QC samples.

7.3 Stock standard solutions – commercially available

7.3.1 Standard stock solutions may be purchased from Spex (XCL-11, XCL-2, XCL-3B, and PLSBR, and STD3).

7.3.2 Label and refer to these solutions as Calibration Standards.

7.3.3 Refer to the QC/Standards Prep for Trace ICP Logbook for details.

7.3.4 Prepare calibration standards bi-monthly.

7.3.5 Calibration standards must be verified using an independent source EPA Quality Control sample immediately after instrument calibration.

7.4 Instrument Check Standards

7.4.1 Prepare calibration blanks using the same acid volumes as used for calibration standards, i.e. 5% HCl and 6% HNO_3.

7.4.2 Prepare and analyze interference check samples (ICSA/ICSAB) to check for spectral interferences.

7.4.3 Analyze a Low Range Standard (LRS), which is at the level of the reporting limit, after the CCB 1.

7.5 Internal Standard

7.5.1 For the TJA Trace analyzer, yttrium is introduced into the sample injection system as an internal standard. See the Standards/QC Preparation for Trace ICP logbook for instructions on making the yttrium internal standard.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All samples must be preserved to a pH <2 with HNO_3.

8.3 Samples must be digested and analyzed within 180 days of collection.

8.4 Soil samples must be stored under refrigeration at 2 - 4^{o} C. Aqueous samples may be stored at ambient temperature.

8.5 After analysis residual digestate is maintained in the ICP room for three months then returned to the Custodian for storage until disposal into the acid waste stream.

9.0 Quality Control

9.1 Instrument Detection Limit

9.1.1 IDLs are determined quarterly for each wavelength used for sample analyses.

9.1.2 IDLs are determined by multiplying by three, the average of the standard deviations obtained on three non-consecutive days (e.g., Monday, Wednesday, and Friday) from the analysis of a standard solution (each analyte in DI water) at a concentration of three to five times the most recently determined IDL with seven consecutive measurements per day.

9.1.3 The IDL for each analyte must be less than the analyte reporting limit.

9.1.3.1 To meet the requirements of the DoD-QSM, the IDL must be less than or equal to the MDL for each analyte.

9.2 Linear Range

9.2.1 The linear range for each ICP-AES instrument must be verified quarterly.

9.2.2 Analyze a high concentration standard, linear range standard (LRS), during a routine analytical sequence. This concentration is the upper limit of the ICP-AES linear range.

9.2.3 The recovery of the LRS must be within plus or minus 5% of the expected value.

9.2.4 Samples with concentrations exceeding the linear range must be diluted.

9.2.5 For DoD-QSM and North Carolina compliance reporting, the linear range is defined by the highest calibration standard.

Note: A high calibration standard that encompasses the concentration of the samples may be used to bracket the sample analyses. This high standard must meet the acceptance criteria in 9.2.3 or 9.2.5.

9.3 Initial Calibration Verification (ICV)

9.3.1 The following solutions are used to verify the calibration:

- ICV1 (Ag, Al, As, Ba, Be, Bi, Cd, Ca, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, B, K, Na, V, and Zn)
- Initial Calibration Blank = (ICB)

9.3.2 The results of the initial calibration verification must agree within 10% of the "true" value for each component. If not, the run must be terminated and the instrument re-calibrated. If the re-calibration does not produce acceptable ICV results, shut down the instrument and request instrument service.

9.3.3 Follow the ICV with the ICB. The laboratory will deviate from the method for calibration blank acceptance criteria by substituting the following: The absolute value of the blank must be less than the statistically-determined laboratory reporting limit (PQL). If not, repeat the blank analysis, and if still above the limit terminate the analysis and recalibrate. If re-calibration does not produce acceptable ICB results, shut down the instrument and request instrument service.

9.3.3.1 To meet the requirements of the DoD-QSM, the analyte concentration in the ICB must be less than 2 times the MDL.

9.4 Continuing Calibration Verification

9.4.1 Verify the calibration every 10 samples and at the end of analytical run.

9.4.2 The results of the check standard must agree within ± 10% of the true value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.4.3 Samples bracketed by passing CCVs are reportable. Any sample associated with a failing CCV must be re-analyzed.

9.5 Continuing Calibration Blank (CCB)

9.5.1 A calibration blank must be analyzed at each wavelength used for analysis immediately after every continuing calibration verification at a frequency of 10% or every 2 hours during the run, whichever is more frequent.

9.5.2 The laboratory will deviate from the method for calibration blank acceptance criteria by substituting the following: The absolute value of the blank (average of the three exposures) must be less than the statistically-determined laboratory reporting limit (PQL). If not, repeat the blank analysis and if still above the limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.5.2.1 To meet the requirements of the DoD-QSM, the analyte concentration in the CCB must be less than 2 times the MDL.

9.6 Interference Check Sample

9.6.1 Analyze the standard reference solutions obtained from EPA or NIST. The interference Check samples (ICSA and ICSAB) are analyzed consecutively at the beginning and the end of the analytical run and analyzed and reported at a frequency no greater than 20 analytical samples.

9.6.2 The ICS must agree within +/-20% of the accepted values. If not terminate the analysis and recalibrate the instrument. If re-calibration does not fix the problem, shut down the instrument and request instrument service.

9.6.2.1 To meet the requirements of the DoD-QSM, the absolute value of the concentration of all the non-spiked analytes in the ICS-A must be < 2 times the MDL (unless the analyte is a verified trace impurity from one of the spiked analytes).

9.7 Low Range Standard (LRS)

9.7.1 Analyze a standard at the reporting limit. This reporting limit standard is used to verify linearity and the ability to detect low levels reliably.

9.7.2 The advisory acceptance criteria for this standard is +/- 50% of the true value.

9.7.2.1 To meet the requirements of the DoD-QSM, the recovery of each analyte in the LRS must fall within +/- 20% of the true value.

9.8 Method Blank

9.8.1 For each digestion batch of up to 20 samples, 10 samples for SC DHEC waters, a method reagent blank is prepared. This blank is used to ascertain whether sample concentrations reflect contamination.

9.8.2 The blank must contain absolute values of analyte concentrations below the reporting limit (PQL). If the blank exceeds the reporting limit, the entire sample batch must be re-prepared along with a new method blank.

If the concentration of any analyte detected in the blank falls between the PQL and IDL, flag the corresponding blank element, if present in any associated samples, with a "B" and report the data with a discussion in the SDG narrative.

9.8.2.1 To meet the requirements of the DoD-QSM, the concentration of the target analytes in the method blank must be < half the reporting limit.

9.8.3 For samples submitted for North Carolina compliance reporting, the concentration of target analytes detected are reported between the MDL and PQL with a "J" flag. Any analytes detected in preparation blanks cannot be greater than one-half the PQL.

9.9 Spiked Sample

9.9.1 One matrix spike/matrix spike duplicate (MS/MSD) pair must be prepared with each SDG. For SC DHEC water samples a duplicate is performed at a frequency of 10%.

9.9.2 If the sample spikes do not meet the acceptance criteria of 75-125% recovery, the corresponding element is flagged with an "N" on the Form 1 to indicate that the element did not recover in the matrix spike acceptably. A post-digestion spike is performed for those elements that fall outside the limit.

9.9.2.1 To meet the requirements of the DoD-QSM, the recoveries of the matrix spike should meet the LCS control limits.

9.9.3 Post Digestion Spike (PDS)

9.9.3.1 Prepare and analyze a PDS, if necessary. The spike addition should result in a value that is 2x the PQL.

9.9.3.2 The PDS results should be within 75% to 125%. If not, the data are reported and discussed in the SDG narrative.

No further action is taken at the bench since the internal standard, yttrium, is employed. The instrument uses the element intensity as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interference, especially in high solids matrices.

9.10 Duplicate Sample Analysis

9.10.1 One duplicate sample should be performed for each SDG. SC DHEC requires a duplicate to be performed at the rate of 10%, and this is satisfied with the use of the MS/MSD and sample duplicate.

9.10.2 If the sample and duplicate do not agree within 20% RPD when the concentration is greater than 10x the IDL, then the affected element is flagged with an "*" to indicate poor duplication of results.

9.11 Laboratory Control Sample

9.11.1 A matrix-specific Laboratory Control Sample (LCS) is prepared with every digestion batch of up to 20 samples. SC DHEC requires a frequency of one per 10 water samples.

9.11.2 The vendor supplies the certified acceptance limits for the solid LCS. The limits of 80-120% are subject to change without notice based upon the current vendor supplied limits. If the LCS falls outside these control limits, the analysis must be terminated, the problem corrected, and the samples associated with that LCS re-digested and reanalyzed.

9.11.2.1 To meet the requirements of the DoD-QSM, the LCS recoveries must be within 80 – 120% of the expected result.

9.12 Serial Dilution

9.12.1 One serial dilution must be performed for each SDG.

9.12.2 Perform a 5x serial dilution on the sample to determine if a chemical or physical interference exists. If the analyte concentration is 50 times or more above the instrument detection limit in the original sample, the serial dilution must then agree within 10%. If not then flag the appropriate elements with an "E" to indicate that an interference exists.

9.12.2.1 To meet the requirements of the DoD-QSM, a PDS must be prepared and analyzed when the serial dilution acceptance critria are not met or all sample analyte concentrations are less than 50 times the MDL.

9.13 Field and/or Equipment Blanks

9.13.1 Samples identified as field and/or equipment blanks should not be used for sample spike, duplicate, or serial dilution analysis. They are supplied to the laboratory at the discretion of the client.

9.14 Contingency

9.14.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.14.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.14.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.14.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 The instrument calibration standards are analyzed at the beginning of the analytical sequence, in the order shown in the Trace ICP run log (Attachment **3**). Calibration is further discussed in the Procedure section that follows.

10.2 All calibration verification standards must meet the criteria specified in section 9.0 before proceeding with sample analysis.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 TJA Trace Analyzer Set Up and Operating Procedure

11.1.1 Cooling Water

11.1.1.1 Turn on the recirculation pump. Thirty (30) psig is required at a flow rate of 700 ml/min.

11.1.2 Argon Supply

11.1.2.1 Open the argon supply to the instrument.

11.1.2.2 A volume rate of 30 SCFH and delivery pressure of 60 psig is required.

11.1.3 Venting System

11.1.3.1 A permanent vent has been installed to provide a proper draft in the torch compartment. A torch fan is permanently installed to force a 22 mph-draft across the end of the plasma torch.

11.1.4 Water in Drain Barrel

11.1.4.1 A hose is connected to the drain outlet from the spray chamber to allow the flow of unused sample waste from the chamber. This drain line acts as a positive seal to the spray chamber; therefore, liquid must always be present in the drain line.

11.2 Power Up (TJA Trace Analyzer)

11.2.1 To start the ICP Trace Analyzer, turn on the video display, the printer, the computer, and the autosampler.

11.2.2 At the "C" prompt, Type "P" and press “ENTER.” This will start the Thermospec™ software and bring up the main menu.

11.2.3 To start the RF generator and the plasma torch, select “SETUP.” Then select “PLASMA CONTROL PANEL” and press “ENTER.” Select “F1.”

11.2.4 The default startup time, during which argon purges the spray chamber and the plasma torch, is 90 seconds. The purge time can be reduced to as little as 20 seconds by pressing “PURGE TIME” if the plasma has been off for less than 15 minutes.

11.2.5 After the purge time and power have been set, the automated plasma startup sequence is initiated by pressing “CONTINUE.”

11.2.6 If the pump and gas have not been started, select “F2” and start the pump rate at 99 RPM and switch the nebulizer gas to “ON.”

11.2.7 The total flush time and rinse time between samples is set at 60 seconds.

11.2.8 Press "LEVELS" if you want to change any of the plasma operating conditions.

11.2.9 The plasma torch should be allowed to run for at least 30 minutes after ignition to reach optimum stability.

11.2 Analysis

11.2.1 Profile

11.2.1.1 Profiling the instrument assures that the optical pathway is clean and in alignment. Emission intensities are essential to accurate data.

11.2.1.2 At the THERMOSPEC main menu, select setup and highlight profile then enter.

11.2.1.3 Begin aspirating 1ppm As standard for analysis.

11.2.1.4 Press F3 to Automatic Profile. When the sample is sufficiently saturating the spray chamber and torch (60 seconds), press "F1", "RUN".

At the end of the integration, a peak graph will be displayed. Note the intensity reading. This will indicate a need to clean the optical path or adjust the alignment.

Also note the peak position on the graph. This value should be between 0.1 and –0.1. If it is not, the spectrum shifter must be adjusted, and the profile rerun. Continue this process until the peak position is acceptable.

11.2.1.5 Discontinue the arsenic when profiling is complete. Exit profile to the main menu.

11.3 Autosampler Table Setup

11.3.1 Obtain all data necessary from each case that will be set up on the run, i.e. preparation logs, log-in chain of custody reports from the LIMS (Attachment 6). The information contained on these sheets will be used to create the autosampler table.

11.3.2 From the main menu of Thermospec, select "OPERATION," and then "Autosampler Setup," then press enter. The software will prompt for an autosampler table name. An existing table can be edited from this point, or a new autosampler table name can be typed in at this point. Press enter. Press F3 "Add Set." Enter the appropriate run parameters to be used. Parameters that should always be entered here are Method Name, Rinse Time (60 seconds), # of Unknowns in this set, Default Limit Check Table Name (Sample). Press the F1 "EdSamples" key. This will allow for sample IDs to be entered.

11.3.3 Under the column titled "Sample Name," enter the CompuChem sample ID. Be certain the proper check table is selected for each analysis that is assigned. The "F" key allow for various modifications to the autosampler table. Use these keys as necessary. It is very important that the proper QC samples be inserted into the table at the necessary frequencies. See section 9.0 for a description of necessary QC and frequencies.

11.3.4 Also, if the "Alt" key on the keyboard is pressed, additional options will appear. One of these options is "EdSampInfo." This option is selected by pressing the "Alt" key and the "F2" key simultaneously. From this screen, additional comments can be added that will appear on the raw data printout. Under the column titled "Comment," the SDG, client sample ID and dilution of the sample should be entered.

11.3.5 When all entries are finished, press the "F9," Done/Keep key to save the table. It is important to remember the name of the table created, as this will be used to start the analysis.

11.3.6 Create a configuration file to store all analytical data. From the main menu of THERMOSPEC select "SETUP" and highlight "CONFIGURATION." Press "Enter" twice then "F9" four times. This will provide the screen to enter the configuration file name. Configuration file names should identify the analyst and the date of the analysis, as well as the instrument being used (P3 or P4). Use the following scheme (iimmdd) to construct the file name. The letter "i" for initials, the "m" for the month, and the letter "d" for the day. If there are more runs in a day, letters of the alphabet are used as a suffix. In this example: P3JCQ922B, identifies a third run on September 22 by analyst JC on instrument P3. JUNK files should be used between analyses to prevent files from being corrupted with other data.

11.3.7 Proceed to analysis. From the main menu of THERMOSPEC select "OPERATION." Highlight "ANALYSIS" then "Enter." Confirm that

the correct method is being used and select the autosampler table to be used. Continue to follow on-screen instructions to initiate the run.

IMPORTANT: If the run should fail at any time, remember to change the configuration file name before restarting the analytical run.

11.4 Standardization and Analysis

11.4.1 Start autosampler table and follow the instructions.

11.4.1.1 Select "Operation" and highlight "Analysis."

11.4.1.2 Enter the correct method of analysis and select the autosampler table for samples to be analyzed.

11.4.2 Solid samples and liquid samples have different matrices due to sample preparation procedures. Calibration standards and quality control standards should be prepared to match the matrix of the samples being analyzed. While constructing autosampler tables, give consideration to the matrix of the samples.

11.4.3 Fill all standards cups and QC cups with the appropriate solutions following the sequence hardcoded on the Trace ICP Run Log (Attachment 5.) The autosampler table provides the information for cup positions. Standards cups positioning may change depending on the number of samples.

Caution: Accuracy of the sequence setup is extremely important. Improper cup positioning will seriously affect analysis to the point that there may not be any salvageable data.

11.4.4 Press "F1" to begin the analytical run.

Caution: Sample racks should be loaded accurately following the rows and positions provided by the autosampler table. Samples in the wrong position can produce erroneous data that may not be detectable.

11.4.5 Although, software drives the instrument operation, the instrument should not be left completely unattended. In the course of the run, matrix spike analyses must be evaluated to determine percent recoveries. Some samples will have concentrations of analytes that are beyond the established linear ranges and will need to be diluted and reanalyzed

within those ranges. For samples submitted for North Carolina compliance reporting, dilutions and re-analyses are required whenever target analytes exceed the highest calibration standard. Serial dilution analyses should be evaluated for proper dilution. Responding to the need for dilutions or a PDS analysis quickly within the same analytical run saves time by not requiring an additional run and improves productivity.

11.4.6 Attention should be given to the QC analyses. Analytes may be found to be outside of their established control limits. The loss of one or more analytes does not necessarily mean the run has ended. Early detection of failed analytes by the analyst will prevent lost time and productivity.

11.4.7 Standards cups have limited capacity and should be filled frequently and monitored at all times.

11.4.8 If the sequence includes an ICSA and ICSAB analysis performed every 20 analytical samples, the structure of the autosampler table must incorporate this requirement. LRS, ICSA and ICSAB are numbered with the analytical samples. If this maximum is exceeded, the run has ended and all data that does not comply is lost. The ICSA and ICSAB must immediately be followed by a CCV/CCB.

Based on client-specific project requirements, additional QC may be run. An example is the CRI standard that is required by the CLP.

11.4.9 The analytical run is not complete until all final paperwork is complete. The run log must be completed by the analyst and the QC/Standards Preparation log completed for the day, as well as completing the internal chain of custody form.

11.5 Transferring Data

11.5.1 At the completion of the analytical run, exit analysis and return to the THERMOSPEC main menu. Change the configuration file name.

11.5.2 Exit THERMOSPEC and return to Desktop. Double-click on the Explorer icon. While in Explorer, open the STATION directory in the left column, and then open the BIN directory. In the right column, find the name of the file from the analytical run that was just finished. Next, find the MARRS directory in the left column and open that directory. Click on the file name in the right column and copy it to the appropriate

directory in MARRS (P3 ICP Trace Files or P4 ICP Trace Files.) Explorer can then be closed.

11.5.3 Transfer the file to the computer used to run the SQUEEZE program. The squeeze function combines two pages of data onto one page. Type "SQZ61E" and follow the instructions to squeeze the data file, and then print the .SQZ file.

11.6 Instrument Shutdown

11.6.1 TJA Trace Analyzer (P3 or P4)

11.6.1.1 Aspirate DI water for approximately one minute.

11.6.1.2 From the MAIN MENU go to SETUP and PLASMA CONTROL PANEL and press enter.

11.6.1.3 Press F7 for PLASMA SHUT off.

11.6.1.4 Remove pump tubing from peristaltic pumps.

12.0 Data Analysis & Calculations

Calculations must be consistent with the Q**uality** C**ontrol** SOP **13.4, "**Numerical Data Reduction**"**.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$Standard\ deviation = \sqrt{\frac{\sum_{i=1}^{n} \left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Calculating Dilutions

If a sample concentration exceeds the linear range of the instrument, a dilution reanalysis must be performed. For samples submitted for DoD-QSM and North Carolina compliance reporting, dilutions and re-analyses are required whenever target analytes exceed the highest calibration standard. Determine a level of dilution that will result in a value within the upper half of the linear range or, for samples submitted for North Carolina compliance reporting, within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

12.8 Concentration

12.8.1 All results for aqueous samples are reported in µg/L as follows.

$$\text{Concentration } (\mu g/L) = \frac{C \times V \times D}{W}$$

Where: C = Concentration (μg/L) from curve
V = Final volume of digestate (L)
W = Volume of sample (L)
D = Dilution factor

12.8.2 All results for solid samples are reported in mg/kg as follows.

12.8.2.1 A separate determination of percent solids must be performed.

12.8.2.2 The concentration determined in the digestate is to be reported on the basis of the dry weight of the sample.

$$\text{Concentration (mg/Kg)} = C * V / W * S$$

Where: C = Concentration (μg/L)
V = Final volume of digestate (L)
W = Weight of wet sample (gm)
S = % Solid / 100
D = Dilution factor

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits, instrument detection limits, and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 6010B

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions

16.5 Q**uality** C**ontrol** SOP **13.6, "**Proper Documentation Procedures**"**

16.6 Q**uality** C**ontrol** SOP **13.4, "**Numerical Data Reduction**"**

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, June 2003, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, **December** 2005, plus revisions.

16.12 CompuChem Quality Manual, Revision 7, Update 1, 12/13/05, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.16 TJA Trace Analyzer Set-up and Operating Manual

16.17 Sample Preparation Procedure –1080, "Digestion Block Preparation of Aqueous Samples for the ICP Determination of Total or Dissolved Metals by SW-846, MCAWW, Standard Methods, and NYSASP"

16.18 Sample Preparation Procedure –240, "Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 and NYSASP."

16.19 Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)"

16.20 Department of Defense Quality Systems Manual of Environmental Laboratories, Final Version 3l, January 2006

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Analyte Concentration Equivalents Arising from Interference at the 100 mg/L Level

17.2 Attachment 2 – Daily Standards/QC Preparation for Trace ICP (P3&P4)

17.3 Attachment 3 – Example Trace ICP Run Log (P3)

17.4 Attachment 4 – Standards Preparation Log for Trace ICP (P3&P4) IEC Studies

17.5 Attachment 5 – Internal Chain of Custody

17.6 Attachment 6 – Reporting Limits

Attachment 1

Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level

Analyte	Wavelength (ηm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	0.21	--	--	_--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.004	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	0.12	--	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	231.604	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	196.026	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybedenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	279.079	--	--	--	--		--	--	--	--	--
Selenium	257.610	0.23	--	--	--	0.09	--	--	--	--	--
Silicon	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Sodium	588.995	--	--		--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--		--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al, Ca, Mg - 1000 mg/L; Ca, Fe - 1000 mg/L; Cr, Cu, Mn, Tl, V - 200 mg/L

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

Attachment 2

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 1 of 4 CompuChem a division of Liberty Analytical Corp. Worksheet 1 P(6)

STANDARDS and REAGENTS TRACEABILITY

Seq. File Name: _____

	Ref #*	Manufacturer	Lot #	Expiration Date
HNO3 [Baker Instra-analyzed Trace-Metals grade]	1M2-176-23	JT Baker	B21039	05/06
HCl [Baker Instra-analyzed Trace-Metals grade]	1M2-176-22	Caledon	54537A	04/06
XCL-11 multi-element standard mix	7M1-33-5	High Purity	516115	07/06
XCL-2 multi-element standard mix	7M1-33-4	High Purity	516116	07/06
XCL-3B multi-element standard mix	7M1-34-17	High Purity	527007	09/06
SPEX-R multi-element standard mix	7M1-35-23	High Purity	607321	03/07
ICP-AES-CRQL-R multi-element standard mix	7M1-32-19	High Purity	510314	05/06
ICV1 multi-element standard mix	7M1-34-26	EPA	1201	01/07
ICSA multi-element standard mix	7M1-35-06	EPA	0503	02/07
ICSB multi-element standard mix	7M1-35-07	EPA	0203	03/07
LRS multi-element standard mix	7M1-34-25	CPI	05J032	04/07
PDS-A multi-element standard mix	7M1-33-19	High Purity	518610	08/06
PDS-B multi-element standard mix	7M1-33-20	High Purity	518610	08/06
ANTIMONY (Sb) 1,000 PPM single-element standard	7M1-33-13	High Purity	510306	01/07
BISMUTH (Bi) 1,000 PPM single-element standard	7M1-33-9	High Purity	506220	01/07
BORON (B) 1,000 PPM single-element standard	7M1-35-04	High Purity	521608	05/07
BORON (B) 1,000 PPM single-element standard (2nd source)	7M1-31-22	CPI	4IG102	05/06
TITANIUM (Ti) 1,000 PPM single-element standard	7M1-33-15	High Purity	515212	01/07
TIN (Sn) 1,000 PPM single-element standard	7M1-33-14	High Purity	511820	01/07
IRON (Fe) 1,000 PPM single-element standard	7M1-33-12	High Purity	509522	01/07
IRON (Fe) 10,000 PPM single-element standard	7M1-35-01	High Purity	529011	05/07
ALUMINUM (Al) 1,000 PPM single-element standard	7M1-33-24	High Purity	518220	02/07
ALUMINUM (Al) 10,000 PPM single-element standard	7M1-34-27	High Purity	524918	05/07
CALCIUM (Ca) 10,000 PPM single-element standard	7M1-35-05	High Purity	522946	05/07
MAGNESIUM (Mg) 10,000 PPM single-element standard	7M1-34-19	High Purity	513903	04/07
MOLYBDENUM (Mo) 1,000 PPM single-element standard	7M1-33-1	High Purity	419721	01/07
ARSENIC (As) 1, 000 PPM single-element standard	7M1-31-12	High Purity	428701	06/06
ZINC (Zn) 1, 000 PPM single-element standard	7M1-34-7	High Purity	515819	03/07
Internal Mix	7M1-34-5	High Purity	523535	09/06

The following standard solutions are applicable only when dissolved samples are analyzed.	Ref #*	Manufacturer	Lot #	Expiration Date
XCL-LCS A multi-element mix	1M2-176-4	High Purity	510803	05/06
XCL-LCS B multi-element mix	1M2-176-5	High Purity	510803	05/06
XCL-20 multi-element mix	1M2-177-8	High Purity	523806	09/06

Entered By: J. BRAUN

Reviewed By: _____ Date: _____

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log

03/29/06:jbb

Attachment 2 (continued)

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 2 of 4 CompuChem a division of Liberty Analytical Corp. Worksheet 1 P(6)

Multi-Element Stock Standard Concentrations:

Stock Standard	Analyte Concentrations
XCL-11 multi-element standard mix	ug/mL: Al:1,000 Cd:1,000 Cr:1,000 Cu:1,000 Pb:1,000 Ni:1,000 Se:1,000 Ag:100 Tl:1,000 Zn:1,000
XCL-2 multi-element standard mix	ug/mL: As:1,000 Ba:1,000 Be:1,000 Co:1,000 Fe:1,000 Mn:1,000 Mo:1,000 V:1,000
XCL-3B multi-element standard mix	ug/mL: K:10,000 Na:10,000
SPEX-R multi-element standard mix	ug/mL: Mo:1,000 Sn:1,000 Bi:1,000 Ti:1,000 B:1,000
ICP-AES-CRQL-R multi-element standard mix	mg/L: Ag:10 Be:5 Cd:5 Co:50 Cr:10 Cu:25 Mn:15 Ni:40 Tl:25 V:50 Zn:60 Pb:10 Se:35 As:10 Sb:60
LRS multi-element standard mix	ug/mL: Al:100 Ba:10 K:1,000 Mg:1,000 Na:2,000 Ca:1,000 Ag:5 Be:5 Cd:5 Co:5 Cr:5 Cu:5 Fe:100 Mn:10 Ni:5 Tl:10 V:20 Zn:20 Pb:3 Se:5 As:10 Mo:5 Sn:20 Bi:50 Ti:40 Sb:10
PDS-A multi-element standard mix	ug/mL: Al:400 Ba:400 Ag:20 Be:10 Cd:10 Co:100 Cr:20 Cu:50 Fe:200 Mn:30 Ni:80 Tl:20 V:100 Zn:40 Pb:6 Se:10 As:20
PDS-B multi-element standard mix	ug/mL: Sb:120
Internal Mix	ug/mL: Li:50,000 Y:250

Entered By: J. BRAUN

Reviewed By: _____ Date: _____

03/29/06 jbb

Attachment 2 (continued)

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 3 of 4 CompuChem a division of Liberty Analytical Corp. Worksheet 1 P(6)

Standard Lot Number*	Preparation instructions	Resultant Analyte Conc. (µg/L)
S0/ICB/CCB–_03_27_06_–_JB	Place in a 500-ml volumetric flask: 100 mL deionized water, _5_ mL concentrated HNO_3, and _25_ mL concentrated HCl. Bring up to volume with deionized water. Prepare as needed	N/A
XCL-11–_03_27_06_–_JB	Place in a 100-ml volumetric flask: 50 mL deionized water, _1_ mL concentrated HNO_3, and _5_ mL concentrated HCl. Pipet 0.2 mL XCL-11 into flask. Bring up to volume with deionized water. Prepare as needed	Al:2,000 Cd:2,000 Cr:2,000 Cu:2,000 Pb:2,000 Ni:2,000 Se:2,000 Ag:200 Tl:2,000 Zn:2,000
XCL-2–_03_27_06_–_JB	Place in a 100-ml volumetric flask: 50 mL deionized water, _1_ mL concentrated HNO_3, and _5_ mL concentrated HCl. Pipet 0.2 mL XCL-2 into flask. Bring up to volume with deionized water. Prepare as needed	As:2,000 Ba:2,000 Be:2,000 Co:2,000 Fe:2,000 Mn:2,000 Mo:2,000 V:2,000
XCL-3B–_03_11_06_–_JB	Place in a 100-ml volumetric flask: 50 mL deionized water, _1_ mL concentrated HNO_3, and _5_ mL concentrated HCl. Pipet 1.0 mL XCL-3B into flask. Bring up to volume with deionized water. Prepare as needed	K:100,000 Na:100,000
PLSBR–_03_23_06_–_JB	Place in a 100-ml volumetric flask: 50 mL deionized water, _1_ mL concentrated HNO_3, and _5_ mL concentrated HCl. From 1,000-ppm standards, pipet 0.2 mL of each: Sn, Bi, Ti, Sb, and B into flask. Bring up to volume with deionized water. Prepare as needed.	Sn:2,000 Bi:2,000 Ti:2,000 Sb:2,000 B:2,000
STD3–_03_27_06_–_JB	Place in a 100-ml volumetric flask: 50 mL deionized water, _1_ mL concentrated HNO_3 and _5_ mL concentrated HCl. From 10,000-ppm standards, pipet 1.0 mL of each: Al, Mg, and Ca, and 0.5 mL Fe. Bring up to volume with deionized water. Prepare as needed	Al:100,000 Mg:100,000 Ca:100,000 Fe:50,000
As–_03_23_06_–_JB	Place in a 100 mL volumetric flask: 50 mL deionized water, 1 mL concentrated HNO_3, and 0.1 mL 1000-ppm As standard. Bring to volume with deionized water.	As:1,000
ICV1–_03_17_06_–_JB	Place in a 200-ml volumetric flask: 100 mL deionized water, _4_ mL concentrated HNO_3, and _20_ mL concentrated HCl. Pipet 40.0 mL ICV1 into flask and 0.4 mL CompuChem SPEX-R. Bring up to volume with deionized water. Transfer to a 500mL Teflon bottle and add 200 mL deionized water.	Al:2,482 Ba:502 K:10,008 Mg:6,003 Na:10,039 Ca:10,180 Ag:495 Be:493 Cd:494 Co:496 Cr:490 Cu:490 Fe:5,107 Mn:495 Ni:492 Tl:1,027 V:501 Zn:1,000 Pb:996 Se:1,005 As:996 Sb:992 Mo:1,000 Sn:1,000 Bi:1,000 Ti:1,000 B:1,000
CCV/CVS1–_03_18_06_–_JB	Place in a 500-ml volumetric flask: 300 mL deionized water, 20 mL concentrated HNO_3, and 100 mL concentrated HCl. From 1,000-ppm standards, pipet 2 mL of each: Sb, Sn, Bi, Ti, and B; and 0.2 mL Zn. From 10,000-ppm standards, pipet 10 mL Ca, 10 mL Mg, 4.8 mL Fe, and 9.8 mL Al. Pipet 2 mL XCL-2, 2 mL XCL-11, and 10 mL XCL-3B. Bring to volume with deionized water. Transfer to a 2 L Teflon bottle. Add 1.5 L deionized water. Prepare as needed.	Al:50,000 Ba:1,000 K:50,000 Mg:50,000 Na:50,000 Ca:50,000 Ag:100 Be:1,000 Cd:1,000 Co:1,000 Cr:1,000 Cu:1,000 Fe:25,000 Mn:1,000 Ni:1,000 Tl:1,000 V:1,000 Zn:1,100 Pb:1,000 Se:1,000 As:1,000 Mo:1,000 Sn:1,000 Bi:1,000 Ti:1,000 Sb:1,000 B:1,000

Standard Solutions Prepared By: _____

Entered By: J. BRAUN

Reviewed By: _____ Date: _____

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

03/29/06:jbb

Attachment 2 (continued)

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 4 of 4 CompuChem a division of Liberty Analytical Corp. Worksheet 1 P(6)

Standard Lot Number*	Preparation instructions	Resultant Analyte Conc. (µg/L)
ICSA– 03 25 06 – JB	Place in a 500-ml volumetric flask: 350 mL of deionized water, 5 mL concentrated HNO_3, and 25 mL concentrated HCl. Pipet 50.0 mL ICSA into flask. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle.	Al:243,900 Ba:2 Ca:234,100 Co:3 Cr:36 Cu:15 Fe:94,900 Mg:248,800 Mn:19 Ni:10 V:1 Zn:39 Pb:5
ICSAB– 03 25 06 – JB	Place in a 500-ml volumetric flask: 50 mL of deionized water, 5 mL concentrated HNO_3 and 25 mL concentrated HCl. Pipet 50.0 mL ICSA and 50.0 mL ICSB into flask. From 1,000-ppm standards, add 0.5 mL of each: Bi, Sn, Ti, and Mo. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle.	Ag:210 Al:248,400 Ba:475 Be:482 Ca:234,300 Cd:916 Co:455 Cr:506 Cu:537 Fe:95,100 Mg:254,100 Mn:483 Ni:930 Tl:96 V:481 Zn:975 Pb:51 Se:51 Sb:585 As:97 Sn:1,000 Bi:1,000 Mo:1,000 Ti:1,000
LRS– 03 02 06 – JB	Place in a 200-ml volumetric flask: 100 mL of deionized water, 2 mL concentrated HNO_3 and 10 mL concentrated HCl. Pipet 0.20 mL LRS and 0.04 mL of B 1,000-ppm standard into flask. Bring up to volume with deionized water.	Al:100 Ba:10 K:1,000 Mg:1,000 Na:2,000 Ca:1,000 Ag:5 Be:5 Cd:5 Co:5 Cr:5 Cu:5 Fe:100 Mn:10 Ni:5 Tl:10 V:20 Zn:20 Pb:3 Se:5 As:10 Mo:5 Sn:20 Bi:50 Ti:40 Sb:10 B:200
EPA-CRI– 03 16 06 – JB	Place in a 200-mL volumetric flask: 100 mL deionized water, 4 mL concentrated HNO_3 and 20 mL concentrated HCl. Pipet 0.40 mL ICP-AES-CRQL-R into flask. From 1,000-ppm standards, add 0.016 mL Bi, 0.100 mL Sn, 0.008 mL Mo, 0.024 mL Ti, and 0.002 mL B. Bring up to volume with deionized water, transfer to a 500mL Teflon bottle and add 200mL deionized water.	Ag:10 Be:5 Cd:5 Co:50 Cr:10 Cu:25 Mn:15 Ni:40 Tl:25 V:50 Zn:60 Pb:10 Se:35 As:10 Mo:20 Sn:250 Bi:40 Ti:60 Sb:60 B:5
COMM-CRI– 01 31 06 – JB	Place in a 200-mL volumetric flask: 100 mL deionized water, 4 mL concentrated HNO_3 and 20 mL concentrated HCl. Pipet 0.40 mL PDS-A and 0.40 mL PDS-B into flask. From 1,000-ppm standards, pipet: 0.016 mL Bi, 0.016 mL Sn, 0.008 mL Mo, 0.024 mL Ti, and 0.200 mL B. Bring up to volume with deionized water, transfer to a 500mL Teflon bottle and add 200mL deionized water.	Al:400 Ba:400 Ag:20 Be:10 Cd:10 Co:100 Cr:20 Cu:50 Fe:200 Mn:30 Ni:80 Tl:20 V:100 Zn:40 Pb:6 Se:10 As:20 Mo:20 Sn:50 Bi:50 Ti:50 Sb:120 B:500
INTSTD– 02 17 06 – JB	Place in a 10 L container: 8 L deionized water, 400 mL concentrated HNO_3, and 200 mL Internal Mix. Fill with deionized water.	Li:1,000,000 Y:5,000

Dissolved Metals only:	Prepared By	Date Prepared	Preparation Instructions
LCS	N/A	N/A	Add 0.1 mL of XCL-LCS solution A and 0.1 mL of XCL-LCS solution B to 9.8 mL acidified water (1% HNO_3 + 5% HCl).
Prep Blank	N/A	N/A	Use acidified water (1% HNO_3 + 5% HCl).
Sample Spike	N/A	N/A	Add 0.1 mL of XCL-20 to 9.9 mL of sample.

Standard Solutions Prepared By: _____

Entered By: J. BRAUN

Reviewed By: _____ Date: _____

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer. 03/29/06 jbb

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Attachment 3

COMPUCHEM a division of Liberty Analytical Corp. LOGBOOK 1 O(4) 63

Trace ICP Run Log (P3) **Part (A) of a three-part form**

Instrument Profile:
Nebulizer pressure:_____
Peak position:_____
Peak intensity:_____
Vernier position:_____

Maintenance:_____
_____
_____
_____
_____

Page__1__of___

Method (circle one): CLP ILM04.1 CLP ILM05.3 SW-846 6010B MCAWW 200.7 _____

Operator:_____ **File name**:__P3_____ **Date/Time**:_____ to _____

SDG	Matrix	Analytes	Comments

Legend		No.	Sample ID	Comments	Action needed
↑	Specified element(s) fail(s) high	1			
↓	Specified element(s) fail(s) low	2			
RR	Re-run sample(s) neat	3			
RR #x	Re-run at a #-fold dilution	4			
#x	Sample run at a #-fold dilution (aliquot of sample diluted with acidified water)	5			
		6			
L	Matrix = leachate	7			
W	Matrix = water	8			
S	Matrix = solid	9			
		10			
			CCV		

No.	Sample ID	Comments	Action needed		CCB		
	S0	Blank		1			
	S	RSTD-1		2			
	S	RSTD-3		3			
	S	RSTD-2		4			
	S	RSTD-4		5			
	ICV1			6			
	ICB			7			
	CRI			8			
	ICSA			9			
	ICSAB			10			
	CCV				CCV		
	CCB				CCB		

COMMENTS

Reviewed By: _____

Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

06/18/06:jbb

Attachment 3 (continued)

COMPUCHEM a division of Liberty Analytical Corp. LOGBOOK 1 O(4) 63

Trace ICP Run Log (P3) **Part (B) of a three-part form** Page_2_of___

File name:_P3________

No.	Sample ID	Comments	Action needed	No.	Sample ID	Comments	Action needed
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		

COMMENTS

Reviewed By: ______________________________

Date: ______________________________

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

06/18/06:jbb

Attachment 3 (continued)

COMPUCHEM a division of Liberty Analytical Corp. LOGBOOK 1 O(4) 63

Trace ICP Run Log (P3) **Part (C) of a three-part form** Page_3_of___

File name:_P3_______

No.	Sample ID	Comments	Action needed	No.	Sample ID	Comments	Action needed
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
	CCV				CCV		
	CCB				CCB		

COMMENTS

Reviewed By: ____________________

Date: ____________________

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

06/18/06:jbb

Attachment 4

Standards Preparation Log for Trace ICP (P3&P4) IEC Studies

1 of 3
Worksheet 1 P(A)

STANDARDS and REAGENTS TRACEABILITY

	Ref #*	Manufacturer	Lot #	Expiration Date
ALUMINUM (Al) 1,000 PPM	7M1-33-24	High Purity	518220	02/07
ALUMINUM (Al) 10,000 PPM	7M1-33-11	High Purity	517431	01/07
ANTIMONY (Sb) 1,000 PPM	7M1-33-13	High Purity	510306	01/07
ARSENIC (As) 1,000 PPM	7M1-31-12	High Purity	428701	06/06
BARIUM (Ba) 1,000 PPM	7M1-34-6	High Purity	513011	03/07
BARIUM (Ba) 10,000 PPM	7M1-31-4	High Purity	420806	06/06
BERYLLIUM (Be) 1,000 PPM	7M1-30-10	High Purity	412720	03/06
BISMUTH (Bi) 1,000 PPM	7M1-33-9	High Purity	506220	01/07
BORON (B) 1,000 PPM	7M1-33-2	High Purity	511014	01/07
BORON (B) 1,000 PPM (2nd source)	7M1-31-22	CPI	4IG102	05/06
CALCIUM (Ca) 10,000 PPM	7M1-33-10	High Purity	513226	01/07
CADMIUM (Cd) 1,000 PPM	7M1-30-13	High Purity	417528	03/06
CESIUM (Cs) 10,000 PPM	7M1-34-11	High Purity	522915	04/07
CHROMIUM (Cr) 1,000 PPM	7M1-31-5	High Purity	424704	06/06
COBALT (Co) 1,000 PPM	7M1-31-6	High Purity	423325	06/06
COPPER (Cu) 1,000 PPM	7M1-30-14	High Purity	421703	03/06
IRON (Fe) 1,000 PPM	7M1-33-12	High Purity	509522	01/07
IRON (Fe) 10,000 PPM	7M1-31-10	High Purity	424536	06/06
LEAD (Pb) 1,000 PPM	7M1-33-22	High Purity	516645	02/07
LITHIUM (Li) 1,000 PPM	7M1-34-12	CPI	04L037	09/06
LITHIUM (Li) 1,000 PPM (2nd source)	7M1-34-13	High Purity	500309	11/06
MAGNESIUM (Mg) 10,000 PPM	7M1-33-3	High Purity	513903	01/07
MANGANESE (Mn) 1,000 PPM	7M1-33-23	High Purity	514421	02/07
MOLYBDENUM (Mo) 1,000 PPM	7M1-33-1	High Purity	419721	01/07
NICKEL (Ni) 1,000 PPM	7M1-31-7	High Purity	426809	06/06
POTASSIUM (K) 10,000 PPM	7M1-30-7	High Purity	414913	03/06
SELENIUM (Se) 1,000 PPM	7M1-34-8	High Purity	518210	03/07
SILVER (Ag) 1,000 PPM	7M1-30-15	High Purity	411001	03/06
SODIUM (Na) 1,000 PPM	7M1-31-8	High Purity	423227	06/06
SODIUM (Na) 10,000 PPM	7M1-30-8	High Purity	419427	06/06
THALLIUM (Tl) 1,000 PPM	7M1-31-9	High Purity	425902	06/06
TIN (Sn) 1,000 PPM	7M1-33-14	High Purity	511820	01/07
TITANIUM (Ti) 1,000 PPM	7M1-33-15	High Purity	515212	01/07
VANADIUM (V) 1,000 PPM	7M1-31-11	High Purity	426130	06/06
YTTRIUM (Y) 1,000 PPM	7M1-28-17	High Purity	405815	11/05
ZINC (Zn) 1,000 PPM	7M1-34-7	High Purity	515819	03/07

Applies to Seq. File(s):_____

Entered By: J. BRAUN

Reviewed By:_____ Date:_____

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log

10/06/05:jbb

Attachment 4 (continued)

Standards Preparation Log for Trace ICP (P3&P4) IEC Studies

2 of 3
Worksheet 1 P(A)

Standard Name	Preparation instructions
Al-500 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 5 mL of 10,000-ppm Al. Bring up to volume with deionized water. Prepare annually.
Ag-2 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.2 mL of 1,000-ppm Ag. Bring up to volume with deionized water. Prepare annually.
As-10 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 1 mL of 1,000-ppm As. Bring up to volume with deionized water. Prepare annually.
B-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm B. Bring up to volume with deionized water. Prepare annually.
Ba-50 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 10,000-ppm Ba. Bring up to volume with deionized water. Prepare annually.
Be-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Be. Bring up to volume with deionized water. Prepare annually.
Bi-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Bi. Bring up to volume with deionized water. Prepare annually.
Ca-500 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 5 mL of 10,000-ppm Ca. Bring up to volume with deionized water. Prepare annually.
Cd-15 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 1.5 mL of 1,000-ppm Cd. Bring up to volume with deionized water. Prepare annually.
Co-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Co. Bring up to volume with deionized water. Prepare annually.
Cr-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Cr. Bring up to volume with deionized water. Prepare annually.
Cu-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Cu. Bring up to volume with deionized water. Prepare annually.
Fe-300 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 3 mL of 10,000-ppm Fe. Bring up to volume with deionized water. Prepare annually.
K-200 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 10,000-ppm K. Bring up to volume with deionized water. Prepare annually.
Li-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Li. Bring up to volume with deionized water. Prepare annually.

Standard Solutions Prepared By:_____

Entered By: _____J. BRAUN_____

Reviewed By:_____ Date:_____

10/06/05:jbb

Attachment 4 (continued)

Standards Preparation Log for Trace ICP (P3&P4) IEC Studies

3 of 3
Worksheet 1 P(A)

Standard Name	Preparation instructions
Mg-500 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 5 mL of 10,000-ppm Mg. Bring up to volume with deionized water. Prepare annually.
Mn-10 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 1 mL of 1,000-ppm Mn. Bring up to volume with deionized water. Prepare annually.
Mo-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Mo. Bring up to volume with deionized water. Prepare annually.
Na-200 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 10,000-ppm Na. Bring up to volume with deionized water. Prepare annually.
Ni-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Ni. Bring up to volume with deionized water. Prepare annually.
Pb-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Pb. Bring up to volume with deionized water. Prepare annually.
Sb-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Sb. Bring up to volume with deionized water. Prepare annually.
Se-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Se. Bring up to volume with deionized water. Prepare annually.
Sn-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Sn. Bring up to volume with deionized water. Prepare annually.
Ti-5 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 0.5 mL of 1,000-ppm Ti. Bring up to volume with deionized water. Prepare annually.
Tl-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Tl. Bring up to volume with deionized water. Prepare annually.
V-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm V. Bring up to volume with deionized water. Prepare annually.
Zn-20 PPM	Place in a 100-ml volumetric flask: 50 ml of deionized water, _1_ml concentrated HNO_3, and _5_ml concentrated HCl. Pipet 2 mL of 1,000-ppm Zn. Bring up to volume with deionized water. Prepare annually.
Li-10 PPM	In a 15-mL beaker, pipet 5 mL acidified water and 5 mL Li-20 PPM. Prepare immediately before analysis.

Standard Solutions Prepared By:_____

Entered By: _____J. BRAUN_____

Reviewed By:_____ Date:_____

10/06/05:jbb

Attachment 5

Internal Chain of Custody
CompuChem a division of Liberty Analytical

ICP ANALYSIS

Case:_____ SDG:_____

Matrix:_____

COC for: raw sample extract digestates leachates aliquots

CompuChem Numbers for Associated Samples

Released by	Received by	Date/Time	Reason

This form generated on: **10/31/2005**

Attachment 6

Analyte	Reporting Limit (Method 6010B)		
	µg/L	mg/Kg	Ohio Vap mg/Kg
Aluminum	200	20.0	
Antimony	10.0	1.0	2.0
Arsenic	10.0	1.0	2.0
Barium	10.0	1.0	20.0
Beryllium	5.0	0.5	
Cadmium	5.0	0.5	
Calcium	5000	500	
Chromium	5.0	0.5	1.0
Cobalt	5.0	0.5	
Copper	5.0	0.5	2.0
Iron	100	10.0	40.0
Lead	3.0	0.3	1.0
Magnesium	5000	500	
Manganese	10.0	1.0	
Nickel	5.0	0.5	4.0
Potassium	5000	500	
Selenium	5.0	0.5	2.0
Silver	5.0	0.5	1.0
Sodium	5000	500	
Thallium	10.0	1.0	
Vanadium	20.0	2.0	
Zinc	20.0	2.0	6.0
Additional analytes			
Bismuth	50.00	5.0	
Molybdenum	5.00	0.5	1.0
Tin	20.00	2.0	3.0
Titanium	40.00	4.0	

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-240 SOP Section #: 3.2.1.5 Revision #: 6

SOP Title: Digestion Block Preparation of ~~Soil~~ Solid Samples for ICP Determination of Total Metals by SW-846 and NYSASP 6/19/06

Effective date: (QA fills in) 3/16/04

- Procedure prepared by: Diane C. Ellmore Date: 3/1/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Linda Carter Date: 3/2/04
- Reason for change: OH VAP compliance
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, Method 3050B; NY State Analytical Services Protocol (NYSASP), June 2000, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 5-8-06

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -240: Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure -240: Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 and NYSASP

1.0 Scope and Application

This method describes the digestion procedure for the preparation of solid samples, including soil, sediment, sludge, and biota, for inductively coupled plasma (ICP) determinations using a 36 or 54 well digestion block. Sample volume and reagent volumes do not differ from the volumes used in the traditional beaker preparation procedure. This procedure is based upon SW-846 Method 3050B and the New York State Analytical Services Protocol (NYSASP). Alternate procedure for Sb, Ba, and Ag is used for Minnesota Pollution Control Authority (MPCA).

Method detection limits (MDLs) and reporting limits are shown in Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A representative 0.95-1.9 gram sample aliquot is digested with repeated additions of nitric acid and hydrogen peroxide. Biota samples must first be homogenized. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is diluted to a final volume of 100 ml.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For some inorganic methods, the reporting limit is based on the MDL, and is usually 4-8 times higher than the MDL.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

For CLP the reporting limit is the Contract Required Detection Limit (CRDL) for inorganics.

3.3 Reporting Units – mg/Kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

4.0 Interferences

4.1 Spectral interference effects can be observed in the determination of trace elements.

4.2 Physical interferences are generally considered to be effects associated with the ICP analysis, especially with samples containing high dissolved solids and/or acid concentrations.

4.3 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects.

4.4 Interferences are highly dependent on matrix type and the specific analyte element. Further detail on managing interferences is found in Instrument Procedure SOP 309, "Inductively Coupled Plasma Atomic Emission Spectroscopy by SW846 and NYSASP."

5.0 Safety

5.1 Care should be taken when handling acids. Always use acids under approved fume hoods. Always pour acid into water.

5.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 In this prep method, the 50 mL cup requires the technician to closely monitor sample reaction to reagents in order to avoid sample overflow.

5.4 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Polypropylene digestion cups - 50 mL

6.2 Ribbed polypropylene watch glasses

6.3 Environmental Express 36 and 54 well digestion blocks

6.4 Whatman No. 41 filter paper or equivalent.

6.5 Analytical balance--capable of accurately weighing to the nearest 0.01 g.

6.6 Glassware – miscellaneous

6.6.1 Except for the preparation of 100 mL Class A volumetric flasks, the labor intensive glassware preparation procedure for inorganics is eliminated when aqueous samples are prepped using a digestion block. See Glassware Preparation Room SOP 10.2, "Preparing Glassware for the Inorganics Laboratory."

6.7 Blender for homogenizing biota samples.

7.0 Reagents & Standards

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (18^{th} and 19^{th} Editions of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Nitric acid (HNO_3) – concentrated, ultra-pure reagent.

7.2.1 All concentrated nitric acid used in preparations should be Baker "Instra-analyzed" (BIA) or an equivalent.

7.2.2 1:1 HNO_3

7.2.2.1 Prepare a 1:1 solution by slowly adding one part acid to one part DI water.

7.3 Hydrochloric acid (HCl) – concentrated, ultra-pure reagent

7.3.1 All concentrated hydrochloric acid used in preparations should be Baker "Instra-analyzed" (BIA) or an equivalent.

7.4 Hydrogen peroxide (30%) - purified and preserved without tin.

7.5 ERA PPT Soil-purchased, certified LCS soil standard.

7.5.1 This standard will be used for OH VAP work.

7.6 EPA LCS Soil-purchased, certified LCS soil standard.

7.6.1 For use in preparing the MCPA LCS

7.7 XCL-20-purchased, certified aqueous standard

7.7.1 For use in spiking the MS/MSD

7.8 XCL-10X sol. A-purchased, certified aqueous standard

7.8.1 For use in preparing the MCPA LCS

7.9 XCL-10X sol. B-purchased, certified aqueous standard

7.9.1 For use in preparing the MCPA LCS

7.10 Dry ice for the homogenization of biota

7.11 All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date three years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.3 Soil samples are stored under refrigeration at 2-4.4° C.

8.4 All samples must be digested and analyzed within 180 days of collection.

8.5 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler at 2-4.4° C for long-term storage and disposal.

8.6 After analysis, the residual digestates are boxed up and stored in the ICP laboratory or Sample Control. After three months they are disposed of in the acid waste stream.

9.0 Quality Control

9.1 Preparation Blank

9.1.1 Prepare a blank for each digestion batch of up to 20 samples.

9.1.2 The preparation blank is prepared using an empty digestion cup to which is added reagents in the same volumes and at the same times as other samples in the batch.

9.2 Laboratory Control Sample

9.2.1 Prepare a laboratory control sample (LCS) for each preparation batch of up to 20 samples.

9.2.2 The LCS is prepared by digesting 1.0 g of an ERA PPT CLP soil in the same manner as the other samples in the batch. **This LCS is used for OH VAP work.**

9.2.2.1 For MPCA samples, prepare two LCSs. One LCS is prepared by digesting one ml of XCL LCS-10X sol. A and one ml of XCL LCS-10X sol. B in the same manner as the other samples in the batch. This LCS is referred to as the LSD.

9.2.2.2 The second LCS is prepared by digesting 1.0 g of EPA LCS solid in the same manner as the other samples in the batch.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for each SDG.

9.3.2 The MS/MSD are prepared by weighing two additional 0.95-1.9 g aliquots of the sample designated as the original, then spiking those aliquots using 1.0 ml of XCL-20. The MS/MSD are digested with the batch of samples. **This MS/MSD is used for OH VAP work.**

9.3.2.1 For MPCA samples, the MS/MSD are prepared by weighing two additional 0.95-1.9 g aliquots of the sample designated as the original, then spiking those aliquots using 1.0 ml of SW2R. The MS/MSD are digested with the batch of samples.

9.4 Duplicate

9.4.1 Prepare a sample duplicate for each SDG. For NC DENR, duplicates are required at a 10% frequency. The duplicate frequency is satisfied with the additional MS/MSD.

9.4.2 The sample duplicate is prepared by weighing an additional 0.95-1.9 g aliquot of the sample designated as the original. The duplicate is digested with the batch samples.

9.5 If samples react violently to peroxide and any sample is lost the sample must be re-prepared.

10.0 Calibration & Standardization

10.1 Ensure that the balance has been calibrated for the day before weighing out samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Turn on the digestion blocks. Refer to the operating instructions for the block (Attachment 2) prior to turning it on for the first time. The set temperature for our elevation is approximately 100°-103° C for the maintenance of 95° C sample temperature.

11.2 Monitor the temperature of each block during sample digestion using a calibrated alcohol thermometer. A cup is set up in each block that contains a thermometer. Periodically the cups need to be re-filled with DI water. Record the temperature on the Metals/Mercury Preparation Log (Attachment 3).

11.3 Request samples to be prepared for the day. Samples are managed and tracked by the Sample Custodian. A properly completed Sample Request Form (Attachment 5) is given to the Sample Custodian. The form accompanies the samples until preparation is completed and they are returned to the Custodian.

11.4 Prepare labels for each sample. The label should contain the laboratory-assigned sample ID and any QC designation.

11.5 Mix the sample well, discard any foreign objects such as sticks, stones/rocks, and leaves. Weigh a representative sample aliquot in the range of 0.95-1.9 gram into a 50-mL digestion cup. Record the weight of the aliquot on the preparation log. Do not target a specific sample weight. The only requirement is that the weight of the aliquot falls within this range. **Prepare associated QC samples as described in section 9.0. QC samples follow the remaining sample preparation instructions.**

11.5.1 If the same biota samples are being prepared for other analyses that require homogenizing, an aliquot of the sample can be taken after homogenizing (but before drying) if needed. If not, proceed to the next step to homogenize.

11.5.2 For biota samples, weigh and record the entire sample. Freeze the sample at –10° C for 2 hours prior to homogenization. Pack the blender below the blades with dry ice. Cut the frozen sample into small pieces and place them in the blender. Cover with dry ice and allow them to sit for one minute to refreeze.

Blend the sample. Pour the sample into a sample container and place the container in the freezer at –10° C. Leave the lid loose so the dry ice can sublimate overnight. The sample will then be ready for the extraction procedure.

Note: Do not air dry the sample.

Note: Biota sample instructions do not pertain to OH VAP work.

11.6 Add 10 ml of 1:1 nitric acid. Swirl the mixture and place a watch glass on the cup. Heat at 95° C for 10-15 minutes without boiling. Remove the sample cup rack from the block. Allow the sample to cool.

11.7 Add 5 ml concentrated nitric acid, and replace the watch glass on the sample cup. Heat for another 30 minutes. Remove the sample cup rack from the block. Allow the samples to cool.

11.8 Repeat step 11.7 once more.

11.9 After the sample has cooled, add three ml of 30% hydrogen peroxide. Wait five minutes and watch the sample for vigorous reaction to the peroxide. Place the sample cup rack back on the digestion block for 15 minutes. Care should be taken to avoid losses due to vigorous effervescence. Continue heating until effervescence subsides, remove from the digestion block, and cool the solution.

11.10 Repeat step 11.9 once more.

11.11 Add 15 ml of DI water to bring the volume in the cup to approximately 40 mL. Add 5 ml concentrated hydrochloric acid. Return the sample rack to the digestion block heat for 15 minutes. Remove the sample cup rack and allow the sample to cool.

11.12 Filter through Whatman #41 filter paper into a 100 ml volumetric flask.

11.12.1 Place Whatman #41 paper in a clean funnel that rests atop a properly cleaned 100 mL volumetric flask (Class A). Prepare the filter and glassware with 1:1 nitric acid followed by a DI water rinse.

11.12.2 Pour the contents of the sample cup into the filter. Thoroughly rinse the insides of the sample cup into the filter using DI water. Ensure that the rinse volume does not cause the sample to rise above the top of the filter.

11.13 Dilute the digestate in the flask to a final volume of 100 ml with DI water.

11.14 For samples requiring improved solubilities and recoveries for antimony, barium, lead, and silver follow these steps.

11.14.1 Add 2.5 ml concentrated HNO_3 and 10 ml concentrated HCl to a 0.95-1.9 gram sample (wet weight) and cover with a watch glass. Place the sample in the heating block and heat for 15 minutes at 95° C.

11.14.2 Filter the digestate through Whatman No. 41 filter paper and collect the filtrate in a 100 ml volumetric flask. Wash the filter paper, while still in the funnel with no more than 5 ml of hot (~95° C) HCl, then with 20 ml of hot (~95° C) DI water. Collect the washings in the same 100 ml volumetric flask.

11.14.3 Remove the filter and residue from the funnel, and place them back in the vessel. Add 5 ml of concentrated HCl, place the vessel back on the heating block, and heat at 95° C +/- 5° C until the filter paper dissolves. Remove the vessel from the heating block and wash the cover and sides with DI water. Filter the residue and collect the filtrate in the same 100 ml volumetric flask. Allow the filtrate to cool, then dilute to volume.

Note: High concentrations of metal salts with temperature sensitive solubilities can result in the formation of precipitates upon cooling of primary and/or secondary filtrates. If precipitation occurs in the flask upon cooling, do not dilute to volume.

11.14.4 If a precipitate forms on the bottom of the flask, add up to 10 ml of concentrated HCl to dissolve the precipitate. After the precipitate is dissolved, dilute to volume with DI water.

11.15 Transfer the tape label from the sample cup to an appropriate 100 mL bottle.

11.16 Transfer the sample from the volumetric flask to the 100 mL bottle and place the bottle in a sample rack.

11.17 Sign and date the internal chain of custody for the digestates to the analysis laboratory.

11.18 Make copies of the prep sheet and relinquish the distilled samples with the prep sheet and ICP chain of custody (Attachment 4) copies to the ICP chemist for analysis. The samples is now ready for analysis following Instrument Procedure 309, "Inductively Coupled Plasma (ICP) Atomic Emission Spectroscopy, Method 6010B."

12.0 Data Analysis & Calculations

12.1 Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.2 Sample results are based upon dry weight of the sample. The dry weight of the sample must be determined according to Sample Preparation Procedure –143, "% Moisture Determination (Undecanted) (EPA CLP, SW846, and NYSASP)." This value used as a correction factor for the determination of the dry weight value.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 1) and precision and accuracy for single analyst (Attachment 6). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Method 3050B.

16.2 New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions

16.3 QCSOP: Proper Documentation Procedures

16.4 QCSOP: Numerical Data Reduction

16.5 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.6 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.7 NELAC Standards, June 2000, plus revisions

16.8 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.9 New York State Environmental Laboratory Approval Program, Certification Manual, June 2000, plus revisions.

16.10 CompuChem Quality Manual, Revision **4**, 1**2**/1**0**/0**3**, plus revisions

16.11 Glassware Preparation Room SOP 10.2, "Preparing Glassware for the Inorganics Laboratory."

16.12 Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples"

16.13 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.14 Instrument Procedure 309, "Inductively Coupled Plasma (ICP) Atomic Emission Spectroscopy, Method 6010B."

16.15 Sample Preparation Procedure –143, "% Moisture Determination (Undecanted) (EPA CLP, SW846, and NYSASP)."

16.16 Instrument Procedure SOP 309, "Inductively Coupled Plasma Atomic Emission Spectroscopy by SW846 and NYSASP."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Method Detection Limit Study

17.2 Attachment 2 – Digestion Block Operating Instructions

17.3 Attachment 3 – Metals/Mercury Preparation Log

17.4 Attachment 4 – Internal Chain of Custody (ICP Analysis)

17.5 Attachment 5 – Internal Chain of Custody (Raw Sample)

17.6 Attachment 6 – Single Analyst Capability Study

Attachment 1

					Digested Soil Metals, Method 3050B/6010B										
Study Dates: January 30, March 11, and April 14, 2003															
Instrument: P4 (Trace)															
Parameter	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Rep#10	Mean	Amount	S.Dev.	MDL	Report Limit
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Aluminum	30.8	31.0	31.6	29.8	30.9	30.3	29.3	30.0	29.2	29.3	30.2	20.0	0.841	2.4	20.0
Antimony	0.658	0.589	0.719	0.507	0.689	0.552	0.653	0.412	0.617	0.480	0.588	1.0	0.0988	0.28	1.0
Arsenic	1.32	1.56	1.31	1.30	1.45	1.46	1.46	1.42	1.63	1.31	1.42	2.0	0.114	0.32	1.0
Barium	0.330	0.449	0.433	0.372	0.354	0.323	0.344	0.328	0.392	0.341	0.367	0.20	0.0445	0.13	1.0
Beryllium	0.0449	0.0422	0.0493	0.0404	0.0442	0.0420	0.0435	0.0401	0.0489	0.0450	0.0440	0.05	0.00315	0.009	0.5
Cadmium	0.190	0.188	0.174	0.190	0.181	0.182	0.159	0.178	0.166	0.167	0.178	0.20	0.0107	0.030	0.5
Calcium	45.8	44.3	46.1	46.0	45.2	44.8	43.0	43.7	44.3	44.5	44.8	20.0	1.01	2.9	500
Chromium	1.25	1.40	1.21	1.24	1.24	1.25	1.23	1.20	1.25	1.24	1.25	1.0	0.0553	0.16	0.5
Cobalt	0.167	0.169	0.183	0.185	0.192	0.187	0.188	0.171	0.160	0.174	0.178	0.20	0.0108	0.031	0.5
Copper	0.258	0.129	0.0854	0.0957	0.113	0.149	0.217	0.122	*	*	0.146	0.20	0.0606	0.18	0.5
Iron **	24.1	24.8	27.7	23.1	23.0	23.8	28.5	23.5	*	*	24.8	20.0	2.13	6.4	10.0
Lead	1.10	0.99	1.00	1.09	1.13	1.01	1.14	1.06	1.06	1.06	1.06	1.0	0.0521	0.15	0.3
Magnesium	11.6	11.5	11.1	11.1	11.4	11.7	11.5	11.8	11.7	11.4	11.5	5.0	0.240	0.68	500
Manganese	0.362	0.338	0.313	0.313	0.355	0.382	0.340	0.355	0.371	0.329	0.346	0.50	0.0233	0.066	1.0
Nickel	0.433	0.456	0.413	0.435	0.428	0.453	0.459	0.505	0.486	0.420	0.449	0.60	0.0292	0.082	0.5
Potassium	54.5	53.6	51.9	54.8	52.6	54.0	50.8	49.9	53.4	49.0	52.5	40.0	2.00	5.6	500
Selenium	1.63	1.40	1.71	1.75	1.46	1.49	1.69	1.64	1.57	1.69	1.60	2.0	0.120	0.34	0.5
Silver	0.201	0.217	0.243	0.221	0.222	0.233	0.235	0.260	0.222	0.221	0.227	0.20	0.0163	0.046	0.5
Sodium	113.8	114.2	123.6	121.5	125.3	118.4	115.1	113.0	119.5	117.6	118.2	100	4.268	12.0	500
Thallium	2.11	1.73	1.96	1.69	1.45	1.89	1.95	1.75	1.85	2.08	1.85	3.0	0.198	0.56	1.0
Vanadium	0.986	1.584	0.960	0.997	0.971	0.993	0.940	0.971	0.979	0.951	1.03	1.0	0.195	0.55	2.0
Zinc	0.690	0.660	0.330	0.328	0.479	0.708	0.537	0.430	0.766	0.415	0.534	1.0	0.162	0.46	2.0
Bismuth	2.92	2.88	2.85	2.61	2.72	2.69	2.81	2.68	2.92	2.61	2.77	3.0	0.122	0.34	5.0
Molybdenum	0.263	0.150	0.225	0.209	0.218	0.189	0.192	0.206	0.133	0.182	0.197	0.50	0.0373	0.11	0.5
Tin	1.95	1.98	1.93	1.69	1.94	1.97	1.91	1.89	1.90	1.89	1.91	1.0	0.0821	0.23	2.0
Titanium	2.72	2.51	2.58	2.51	2.61	2.56	2.51	2.52	2.64	2.59	2.58	2.0	0.0685	0.19	4.0
* Replicate eliminated by Dixon outlier test															

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 the reporting limit.

Attachment 2

Operating Instructions

Catalog Number:

36 well Digestion Block	SC100
Polypropylene Digestion Cups,500/pk.	SC500
18 Cup Tray	SC200

The HOT BLOCK

Specifications

Size: 15" X 15"
Voltage: 120 VAC
Well Size: 3.14" X 2.75"
Max. Cup Temperature: 130°C
Temperature Range: Ambient - 165°C
Controller: Ogden ETR -3020
Temperature Stability: ± 0.1°C
Temperature Uniformity: ± 1°C
Time to Heatup (empty): 13 minutes
Time to Heatup (loaded): 25 minutes
Weight: 43 lb.
Current: 5 amps maximum
Cup Capacity: 50mL

Environmental Express, Ltd.
490 Wando Park Blvd.
Mt. Pleasant, SC 29464
800-343-5319 (o)
843-881-3964 (f)
www. envexp.com

The Environmental Express® Hot Block™ provides an elegant and efficient method of digesting and storing water, wastewater, soil and sludge samples for metals analysis. This innovative system allows samples to be digested in a corrosion free environment. In addition, samples are handled in a small area with minimal radiant heat loss.

Construction
The Hot Block contains almost no metal components. Only the thermocouple (buried and sealed in the graphite), and the controller contacts (sealed with acrylic), are metal. A nitride coating prevents oxidative agents from attacking the integrity of graphite surfaces, and standard digestion agents such as HCL and HNO3 demonstrate no adverse effect on graphite or the fire resistant, box construction material, Kydex®. Box fasteners are PVC. The internal assembly is encased with 1" of thermal insulation.

Location
Locate the Hot Block under a fume hood with a minimum face velocity of 100 fpm, and allow a minimum of 2" of space on all sides.

Temperature Setting
The preset factory "Control Point" temperature is 115°C. Tests have shown that acid solutions in the polypropylene digestion cups with a Control Point temperature of 115°C will achieve 95°C at 760mm of Mercury (sea level). If digestions are being performed at higher elevations it may be necessary to reduce the set point temperature.
Note: The maximum use temperature of the polypropylene cup is 130° C.

To Adjust Temperature
(1) Turn the Hot Block on and wait until the display shows the current block temperature.

(2) Press the left arrow key. The display will show the Control Point temperature. The flashing digit can be changed with the up or down arrow keys.
(3) To change to another flashing digit, press the left arrow key.
(4) When the proper Control Point is reached, press the set key to return to the current temperature display.

Temperature Limit
The controller of the Hot Block has an upper control limit of 150°C. This upper control limit may be changed if desired (see the accompanying controller manual). There is a non-serviceable safety fuse set at 168°C. If the block temperature rises above 168°C, this fuse will melt. (A complete guide to the controller is included with your Hot Block). It is important to note that different liquids may achieve different temperature at the same Hot Block temperature setting. This is due to different vapor pressures of different liquids. When performing standard acid digestions of environmental samples, samples with high oil content will achieve higher temperatures than aqueous samples.

Maintenance
The Hot Block has no serviceable parts. Any service inquiries should be directed to Environmental Express. The unit should be cleaned regularly with water to remove acid residue. Acid that is spilled directly in the digestion wells should be neutralized and removed.

Warranty
This product is warranted against defects in materials and workmanship when used in accordance with the applicable instructions for a period of one year from the date of purchase. The warranty remedy is limited to product repair. If Environmental Express is unable to repair the Hot Block, the customer may, at his/her option receive a replacement unit or a full refund. **Operating the Hot Block at temperatures higher than 160°C will void the warranty.**

Note: Attachment is subject to change without notice.

Attachment 3

ICP Metals/Mercury Preparation Log CompuChem a division of Liberty Analytical Corp.

Metals Method (circle one): CLP(ILM04.0/ILM04.1) 200.7 3010A 3020A 3050B 3005A Sample Prep Included 3030C

Mercury Method (circle one): **MCAWW:** 245.1 245.5 **ILM04.0/ILM04.1** 245.1-M 245.5-M **SW846:** 7470A 7471A

SPP -074/-162/-229/ -239/-240/-1079/-1080

Case/SDG: Prepared by: Date: PREP LOG GENERATED ON:

#	CCN (Lab ID)	Client ID	Date Rec'd	ICAP Initial mL g	ICAP Final (mL)	HG Initial mL g	HG Final (mL)	Description Before	Description After (ICAP)	Description After (HG)	pH
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21	Sample Spike :							Ref CCN # : ( )		Sample Spike(SS) Ref:	
22	Sample Spike Dup. :							Ref CCN # : ( )		ICAP:	
23	Duplicate Sample :							Ref CCN # : ( )			
24	Lab Control Sample :							Reviewed by:_____		HG:	
25	Prep Blank :							Date:_____			
Reagent Manufacturer & Lot #'s:								Time Hg in water bath:		LCS Ref:	
								Time Hg out water bath:		ICAP:	
								water bath temp:			
								Hot block temp :		HG:	

NOTE: All standards and QC for mercury were prepared at the same time as the associated samples.
Refer to the attached Standard Preparation Logbook, with date matching the above preparation date, for preparation method and instruction.

Note: Attachment is subject to change without notice.

Attachment 4

Compuchem a Division of Liberty Analytical
Internal Chain of Custody

ICP ANALYSIS

Project:_____

Case:_____ SDG:_____

Matrix:_____

COC for: raw sample extract digestates leachates aliquots other(explain):_____

CompuChem Numbers for Associated Samples

Released by	Received by	Date	Reason

This form generated on:

Note: Attachment is subject to change without notice.

Attachment 5

CompuChem a Division of Liberty Analytical
Internal Chain of Custody

RAW SAMPLE

Laboratory:_____ Requested By:_____

Water_____ Soil_____ P.E._____ Date:_____

EPA_____ Commercial_____ Shift: 1 2 3

This form generated on:

	CCN	Receipt Date	Preservation	Analysis Parameter	Bottle Number (receiving use only)
1				METALS HG	of
2				METALS HG	of
3				METALS HG	of
4				METALS HG	of
5				METALS HG	of
6				METALS HG	of
7				METALS HG	of
8				METALS HG	of
9				METALS HG	of
10				METALS HG	of
11				METALS HG	of
12				METALS HG	of
13				METALS HG	of
14				METALS HG	of
15				METALS HG	of
16				METALS HG	of
17				METALS HG	of
18				METALS HG	of
19				METALS HG	of
20				METALS HG	of

Relinquished By:_____ Date:_____ Received By:_____ Date:_____

Relinquished By:_____ Date:_____ Received By:_____ Date:_____

Relinquished By:_____ Date:_____ Received By:_____ Date:_____

Note: Attachment is subject to change without notice.

Attachment 6

Laboratory Name/North Carolina Certificate Number: CompuChem/79											
Analyst: Joe Mohn											
Study date: 12/7/01											
Instrument: P3 (Trace)											
Method: Soil ICP Metals by SW846 Method 3050B											
Parameter	TruVal	Rep #1	Rep #2	Rep #3	Rep #4	Mean	ERA/SOP range	Mean	SD(n-1)	EPA	RSD
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/kg	% R	mg/kg	SD	%
Aluminum	8830	6961	7213	6925	7116	7054	3780-13900	80	135.0	NA	1.9
Antimony	68.9	38	42	37	41	40	18.8-119	58	2.6	NA	6.6
Arsenic	136	129	131	131	128	130	101-171	96	1.5	NA	1.1
Barium	124	119	120	121	130	122	95.3-152	99	4.8	NA	3.9
Beryllium	95	90	91	94	89	91	74.7-116	95	2.0	NA	2.2
Cadmium	118	109	111	111	107	110	90.4-145	93	1.9	NA	1.7
Calcium	11500	10997	10999	10814	10685	10874	8590-14400	95	152.8	NA	1.4
Chromium	89	80	80	82	77	80	71.3-107	89	1.9	NA	2.4
Cobalt	110	105	106	109	104	106	87.2-132	96	2.0	NA	1.9
Copper	117	120	120	127	117	121	95.7-138	103	4.3	NA	3.6
Iron	13700	10275	11087	10491	10961	10703	8340-19100	78	383.8	NA	3.6
Lead	138	130	135	132	126	131	105-170	95	4.0	NA	3.1
Magnesium	3040	2498	2550	2583	2507	2535	2150-3930	83	39.8	NA	1.6
Manganese	341	298	291	291	310	298	272-409	87	8.9	NA	3.0
Nickel	156	147	149	150	146	148	122-190	95	2.0	NA	1.3
Potassium	3430	3221	3231	3243	3262	3239	2670-4190	94	17.6	NA	0.54
Selenium	88	91	92	92	90	91	64.9-110	104	1.0	NA	1.1
Silver	119	114	120	122	118	119	88.8-150	100	3.3	NA	2.8
Sodium	853	656	699	669	688	678	578-1130	79	19.3	NA	2.8
Thallium	139	135	136	134	132	134	79.6-199	97	1.7	NA	1.3
Vanadium	79	69	69	71	69	69	54.0-104	88	1.0	NA	1.4
Zinc	66	53	55.80	59	55	56	42.9-89.1	84	2.3	NA	4.2

Note: Attachment is subject to change without notice.

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure __√__ outdated procedure (archive) _____

- Procedure Code: SPP-143 SOP Section #: 2.8.2 Revision #: 6 1/12/06

SOP Title: Effective date: (QA fills in)

Decanted Percent Moisture in Soil/Sediment/Sludge by CLP, SW-846, and NYSASP 9/6/02

- Procedure prepared by: Date:

Diane Ellmore 9/5/02

- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date:

9.6.2

- Reason for change: Updated format and added table of contents

- This procedure meets the requirements of the following approved method references:

New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions; NELAC Standards, June 2000, plus revisions; SW-846, 3rd Edition, Update III; US EPA CLP SOW OLM04.2, OLM04.3, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above) APPROVED

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 4/1/03

Annual Review—Signature: _____ Date: 1/15/04

Annual Review—Signature: _____ Date: 2/16/05

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SOP DOCUMENTATION FORM - ADDENDUM
ANNUAL REVIEW SIGNATURES

This form documents annual review signatures. This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review.

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SOP-143 SOP Section #: 2.8.2 Revision #: 5

SOP Title: Decanted % Moisture by CLP, SW 846 + NYSASP

Effective date: (QA fills in) 9/6/02

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed. The first three annual reviews will appear on the primary SOP Documentation Form. Additional annual reviews are recorded below.

Annual Review—Signature: _____ Date: 7/5/06

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -164: Decanted Percent Moisture in **Soil/Sediment**/Sludge by EPA CLP, NYSASP, and SW-846

Table of Contents

Sample Preparation Procedure -164: Decanted Percent Moisture in **Soil/Sediment**/Sludge by EPA CLP, NYSASP, and SW-846

1.0 Summary of Method

This method is applied to samples designated for semivolatile and/or pesticide/PCB analyses, which contain standing water. Percent solids and dry weight may also be determined from this method.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

A well-mixed portion of the solid sample, from which standing water has been decanted, is added to a pre-weighed weighing dish. The sample is placed in a drying oven (105°C) overnight, until a constant weight is obtained. It is then cooled in a desiccator before weighing.

3.0 Definitions

3.1 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each **7** calendar day period during which field samples in a case are received (**14** calendar days **if requested by the client**) beginning with the receipt of the first sample.

3.2 LIMS – Laboratory Information Management System

3.3 CLP – Contract Laboratory Program

3.4 SOW – Statement of Work

4.0 Interferences

N/A

5.0 Safety

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Aluminum weighing dish capable of holding 10-25 g of sample

6.2 Drying oven capable of reading and maintaining 105°C for 24 hours

6.3 Top loading balance capable of reading 0.01 g

6.4 Spatula

6.5 Desiccator

7.0 Reagents & Standards

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 Samples must be weighed to a constant weight if they are not able to be dried overnight, as in the case of quick turnaround samples. The procedure in Section 11.5 below is followed in these instances.

9.2 Balances must be calibrated before use. After use the balance must be cleaned of debris and residual sample, if any has spilled.

10.0 Calibration & Standardization

10.1 Ensure balance has been calibrated for the day before weighing samples. Refer to **Quality Control** SOP **13.16**, "Top Loading Balance Calibration & Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Decant and discard any water layer on a sediment sample. Mix sample thoroughly to achieve homogeneity. Discard any foreign objects such as sticks, stones, and leaves. Allow the sample to come to room temperature.

11.2 Calibrate the balance and record the calibration into the appropriate logbook. Zero the balance before all weighings. Record all weights to 0.01 g. Place an empty weighing dish on the balance pan and record the weight of the dish on the laboratory worksheet (Attachment 1). See attached example (Attachmend 2) of computer generated worksheet.

11.3 Transfer 5 - 10 g of thoroughly mixed sample to the weighing dish utilizing a spatula. Record the weight of the dish plus wet sample on the laboratory worksheet, in the "Wt. of Wet Sample" column.

11.4 Place the weighing dish (plus sample) in a drying oven set at 105°C. Samples are usually dried overnight and then placed in a desiccator to cool before weighing. Record the weight of the dish plus dry sample in the "Wt. of Dry Sample" column.

11.5 A sample requiring a % moisture/% solids/dry weight determination within the same day is required to be dried to constant weight. This is accomplished by placing the dish plus sample into the 105°C oven for a minimum of two hours. After that time, the sample is removed from the oven and placed in a desiccator to cool. The sample is then weighed and the weight of the dish plus dry sample is placed in the first half of the "Wt. of Dry Sample" column. The dish plus sample is placed back in the oven for an additional one hour, removed from the oven, placed in a desiccator to cool, and re-weighed. Constant weight is achieved when both entries in the "Wt. of Dry Sample" column are the same or the weighings agree within ± 0.02g. If constant weight cannot be achieved after two drying

cycles (dry, desiccate, weigh), repeat the drying cycle until constant weight can be achieved.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 The information contained on the attached worksheet is entered into the LIMS where calculations are performed to determine 1) the Dry Weight Factor, 2) the Percent Moisture, and 3) the Percent Solids.

12.1.1 Dry Weight Factor =

$$\frac{\text{Weight of dish plus wet sample - Weight Empty Dish}}{\text{Weight of dish plus dry sample - Weight Empty Dish}}$$

12.1.2 % Moisture =

$$\frac{\text{Wet sample weight - dry sample weight}}{\text{Wet sample weight}}$$

12.1.3 % Solids =100 - % Moisture

13.0 Method Performance

N/A

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW OLM04.2, **OLM04.3,** plus revisions

16.2 New York State Analytical Services Protocol (NYSASP), June 200**, plus revision**

16.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96.

16.4 QCSOP: Proper Documentation Procedures

16.5 QCSOP: Numerical Data Reduction

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, **June 2000**, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.11 CompuChem Quality Manual, Revision **3, 12/19/02, plus revisions**

16.12 **Quality Control** SOP **13.16**, "Top Loading Balance Calibration & Maintenance."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Dry weight worksheet

Attachment 1

CompuChem a division of Liberty Analytical Corp. Worksheet

Decanted Dry Weight Worksheet

QUEUE #113 SPP-164

Date Assigned: _____ Assigned To: _____

Date Completed: _____ Employee ID #: _____

	Sample Number	Weight of Container (0.00g)	Weight of Container & Wet Sample (0.00)	Weight of Container & Dry Sample (0.00g)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

6/25/01:dce

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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SOP-818 SOP Section #: 2.7.6 Revision #: 6

SOP Title: Synthetic Precipitation Leaching Procedure by SW-846

Effective date: (QA fills in) 4/16/03

- Procedure prepared by: _____ Date: 4/3/03
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane Ellmore Date: 4/16/03
- Reason for change: Annual Review
- This procedure meets the requirements of the following approved method references: SW846, 3rd Edition, Update III, 12/96, Method 1312; NYSASP, June 2000, plus revisions

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** _____ Date: _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 1/15/04

Annual Review—Signature: _____ Date: 3/9/05

Annual Review—Signature: _____ Date: 7/5/06

Sample Preparation Procedure -818: Synthetic Precipitation Leaching Procedure by SW-846

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Sample Preparation Procedure -818: Synthetic Precipitation Leaching Procedure by SW-846

1.0 Scope and Application

Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in samples of soils, wastes, and wastewaters. Leachates generated from the application of this method are assessed for inorganic, volatile, semivolatile, and pesticide analytes using SW846 analytical methods.

The range of measurement is based on the individual analytical methods. Limits of detection are not applicable for Method 1312.

If a total analysis of the soil, waste, or wastewaters demonstrates that individual analytes are not present, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, Method 1312 need not be performed.

If an analysis of any one of the liquid fractions of the 1312 extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from other fractions of the extract, the concentrations would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

If an analysis of an extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, the extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

Staff members performing the procedures described in this standard operating procedure (SOP) are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

For liquid samples (i.e., those containing less than 0.5% dry solid material), the sample, after filtration through a 0.6-0.8 µm glass fiber filter, is defined as the 1312 extract.

For soil samples containing greater than 0.5% solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Definitions

3.1 An SDG is defined by the following, whichever is more frequent:

each 20 field samples received within a case, or

each **7** calendar day period during which field samples in a case are received (**14** calendar days **if requested by the client**) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers **(US ACE) and South Carolina Department of Health and Environmental Control (SC DHEC)** do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **for US ACE and 10% for SC DHEC**. If samples are batched together from different sites, project-specific QC must be processed.

3.2 SPLP - Synthetic Precipitation Leaching Procedure

3.3 ZHE – Zero Headspace Extraction

3.4 Percent solids - that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

3.5 PSR = Particle size reduction (is required unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension, i.e. capable of passing through a 9.5 mm [0.375 inch] standard sieve.)

4.0 Interferences

4.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods in SW-846. All reagents and materials used in preparing samples by Method 1312 must be demonstrated to be free from contaminants and interferences by processing a method blank with every batch of samples prepared. The frequency of method blanks is 1 blank for every 20 samples prepared or every batch, whichever is the most frequent.

5.0 Safety

5.1 At a minimum, gloves, safety glasses, and lab coats must be worn at all times when processing samples by Method 1312. In addition dust masks or respirators, full face shields, exhaust hoods, safety showers, fire extinguishers, laboratory safety procedures, and training in the use of all safety equipment and procedures must be provided for every individual performing this or any other extraction or analytical procedure.

5.2 The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Agitation apparatus

6.1.1 The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm. Any suitable device that rotates end-over-end is acceptable.

6.1.2 Verify the agitation apparatus rotates at 30 rpm ± 2. To calculate rpm, count the rpm for 30 seconds and multiply the number by two. Document that you checked it by recording this number in the designated space on the Synthetic Precipitation Leaching Procedure Worksheet (Attachment 1).

6.2 Bottle extract vessel

6.2.1 The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste. It is

recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles other than polytetrafluoroethylene cannot be used if organics are to be investigated.

6.3 Nonvolatile vessels

6.3.1 When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in the vessel.

6.4 Zero Headspace Extraction (ZHE) vessel

6.4.1 This device is normally used to evaluate the mobility of volatile target analytes. The vessel allows for initial liquid/solid separation, extraction and final extract filtration without having to open the vessel.

6.4.2 The internal volume of the vessel is 500 + mls. The devices contain Viton O-rings for closure. Gas pressure is used to activate the ZHE piston

6.5 Filtration devices

6.5.1 When the waste is evaluated for nonvolatile analytes, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered.

6.5.2 These devices must have a minimum internal volume of 300 ml and accommodate a minimum filter size of 47 mm.

6.5.3 Filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended.

6.5.4 Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

6.6 Materials of Construction

6.6.1 Extraction vessels and filtration devices are made of inert materials that will not leach or absorb sample components.

6.6.1 Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components.

6.6.2 Devices made of high-density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals.

6.6.3 Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

6.7 Filters

6.7.1 Filters are made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6-0.8 µm or equivalent.

6.7.2 Pre-filters must not be used.

6.7.3 When evaluating the mobility of metals, filters must be acid-washed before use by rinsing with 1N nitric acid (HNO_3), followed by three consecutive rinses with DI water (a minimum of 1-L per rinse is recommended).

6.7.4 Glass fiber filters are fragile and should be handled with care.

6.8 pH Meters

6.8.1 The meter should be accurate to ± 0.05 units at 25°C.

6.9 Laboratory balance

6.9.1 Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

6.10 Beaker or Erlenmeyer flask - 500 ml.

6.11 Watchglass - appropriate diameter to cover beaker or Erlenmeyer flask.

6.12 Magnetic stirrer

6.13 ZHE Extract Collection Devices

6.13.1 Tedlar ® bags are most often used to collect any initial liquid phase and the final extract.

6.13.2 Glass, stainless steel or PTEE gas-tight syringes may also be used.

6.14 ZHE Extraction Fluid Transfer Device

6.14.1 A pressurized stainless steel canister is used to transfer the extraction fluid (DI water) into the ZHE.

6.15 Oven

6.15.1 The laboratory drying oven should be capable of maintaining a temperature of 100°± 5°C.

6.15.2 The oven temperature must be read and recorded on the logbook for the unit on each day of use.

7.0 Reagents & Standards

7.1 Reagent water - All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm (**20th** edition of Standards Methods, Method 1080), and referred throughout this SOP as DI or reagent water. This water would have the following analyte-specific characteristics:

7.1.1 Inorganic Analytes: Water that is generated by any method that would achieve the performance standards for ASTM Type II water. The analytes of concern must be no higher than the highest of either the detection limit, five percent of the regulatory level for that analyte, or five percent of the measured concentration in the sample.

7.1.2 Volatile Analytes: Water in which an interferent is not observed at the method detection limit of the compounds of interest. Organic-free water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free DI water.

7.1.2.1 Organic-free water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hour. The analytes of concern must be no higher than the highest of either the detection limit, five percent of the regulatory level for that analyte, or five percent of the measured concentration in the

sample. Exceptions may have to be taken for the common laboratory solvents methylene chloride and acetone.

7.1.3 Semivolatile Analytes: Water in which an interferent is not observed at the method detection limit of the compounds of interest. Organic-free water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. The analytes of concern must be no higher than the highest of either the detection limit, or five percent of the regulatory level for that analyte, or five percent of the measured concentration in the sample. Exceptions may have to be taken for the phthalate esters.

7.2 Sulfuric acid/nitric acid H_2SO_4/HNO_3 (60/40 weight percent mixture)

7.2.1 Cautiously and slowly mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.3 Extraction Fluids

These fluids must be made fresh daily and recorded in the Synthetic Precipitation Leaching Procedure Worksheet (Attachment 1). Include the lot number of the pH buffer solutions being used.

The pH should be checked before use to ensure that the fluid is made up accurately and should be monitored frequently for impurities.

7.3.1 Extraction Fluid #1

7.3.1.1 To prepare the fluid, add the 60/40 weight percent mixture of H_2SO_4/HNO_3 to DI water until the pH is 4.20 ± 0.05.

Note**:** Solutions are not buffered and the exact pH may not be attained.

7.3.1.2 Extraction fluid #1 is used to determine the leachability of the soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters. This fluid must be used for samples specified from EPA Region V.

7.3.2 Extraction Fluid #2

7.3.2.1 This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids to DI water until the pH is 5.00 ± 0.05.

7.3.2.2 The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

7.3.3 Extraction Fluid #3

7.3.3.1 This fluid is DI water and is used to determine the leachability of cyanide and volatile organics.

8.0 Sample Collection, Preservation, and Storage

8.1 All samples are collected using an appropriate sampling plan.

8.2 There may be requirements on the minimal size of the field sample depending on the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

8.3 Preservatives are not added to samples before extraction.

8.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5 When the sample is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples must be collected and stored in a manner that prevents the loss of volatile analytes (e.g., samples should be collected in Teflon™-lined septum capped vials and stored at 4° C. Samples should be opened only immediately prior to extraction.

8.6 SPLP extracts should be prepared for analysis and analyzed as soon as possible after extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid (HNO_3)to a pH <2, unless precipitation occurs. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into

contact with the atmosphere to prevent losses. If they need to be stored, even for a short time, storage must be at 4°C.

8.7 Samples must undergo the SPLP within the following holding time periods. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

Sample Maximum Holding Times (Days)

From:	Field Collection	1312 Extraction	Preparative Extraction	Total Elapsed Time
To:	1312 Extraction	Preparative Extraction	Analytical Analysis	
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury (Hg)	28	NA	28	56
Metal, except Hg	180	NA	180	360

NA= Not Applicable

9.0 Quality Control

9.1 Blank

9.1.1 At least one blank will be analyzed by the same extraction fluid as used for the samples for every 20 extractions that have been conducted in an extraction vessel. The blank is prepared by the SPLP extraction procedure with the samples.

9.2 Duplicates

9.2.1 Duplicates are prepared at a minimum of one in 20 samples. The duplicate is taken through the SPLP extraction procedure with samples.

9.3 Matrix Spike

9.3.1 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the

regulatory level and the data are being used only to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

9.3.2 The matrix spike standard is to be added after filtration of the 1312 extract and before preservation. Matrix spike standard should not be added before 1312 extraction of the sample.

9.3.3 In most cases, matrix spike levels should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one-half the regulatory level, the spike concentration may be as low as one-half of the analyte concentration, but may not be less than five times the method detection limit. To avoid differences in matrix effects, the matrix spikes must be added to the nominal volume of the 1312 extract as that which was analyzed for the unspiked sample.

9.3.4 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the 1312 when the recovery of the matrix spike is below the expected analytical method performance.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS) is generated during the preparative extraction procedure for the specific fraction. The purpose of the LCS is to monitor the performance of the analytical method.

10.0 Calibration and Standardization

10.1 The pH meter must be calibrated with each day of use and recorded in the pH Meter Logbook (Attachment 2). Calibration must be performed according to Section 10.0 in Sample Preparation Procedure SOP –157, "pH Measurement for Solid Samples by EPA CLP and NYSASP."

10.2 The balance must be calibrated daily and recorded in the balance logbook (Attachment 3) according to the procedures discussed in QC SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. Document all information on the Synthetic Precipitation Leaching Procedure Worksheet (attachment 3).

11.1 Preliminary Evaluations

Perform preliminary 1312 evaluations on a minimum of a 100-g aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include:

- determining the percent solids (section 11.1.1)
- determining whether the waste contains insignificant solids and is, therefore, its own extract after filtration (section 11.1.2)
- determining whether the solid portion of the waste requires particle size reduction (section 11.1.3).
- determining which extraction fluid to use (section 11.1.4)

11.1.1 Preliminary determination of percent solids

11.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solids), weigh out a representative subsample (100 g minimum) and proceed to Section 11.1.3.

11.1.1.2 If the subsample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device discussed in Section 6.6 above and is outlined in Section 11.1.1.3 through 11.1.1.9.

11.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

11.1.1.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

11.1.1.5 Weigh out a subsample of the waste (100 g minimum) and record the weight.

11.1.1.6 Allow slurries to stand for no longer than 2 hours to permit the solid phase to settle. Samples that settle slowly may be centrifuged before filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

11.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If the sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight to determine the weight of the sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point if not reached under 10 psi, and if no individual liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to maximum of 50 psi. After each incremental increase of 10-psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e. filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

11.1.1.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

11.1.1.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase from the weight of the sample by subtracting the weight of the liquid phase from the weight of the total sample. See Section 12.0, Data Analysis and Calculations, for details. Record the weight of the liquid phase and solid phases.

11.1.2 If the percent solids determined is equal to or greater that 0.5%, then proceed either to Section 11.1.3 to determine whether the solid material requires particle size reduction or continue with the next step if you notice that a small amount of the filtrate is entrained in wetting the filter. If the percent solids is less than 0.5%, then proceed to Section 11.2.9 if the nonvolatile analysis is to be performed, and to step 11.3 with a fresh portion of the waste if the volatile 1312 analysis is to be performed.

11.1.2.1 Remove the solid phase and filter from the filtration apparatus.

11.1.2.2 Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value within ± 1%. Record the final weight.

Note: Be careful to ensure that the subject solid will not flash upon heating. The drying oven should be vented to a hood or other appropriate device.

11.1.2.3 Calculate the percent dry solids. See section 12.0, Data Analysis and Calculations, for details.

If the percent dry solids is less than 0.5%, then proceed to Section 11.2.9, if the nonvolatile analysis is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile analysis is to be performed, return to the beginning of Section 11.1 and, with a fresh portion of sample, determine whether particle size reduction is necessary (Section 11.1.3).

11.1.3 Preliminary determination of whether the sample requires particle-size reduction (particle-size is reduced during this step)

11.1.3.1 Using the solid portion of the sample, evaluate the solid for particle size. Particle-size reduction is required unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5-mm (0.375 inch) standard sieve. If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above.

Note: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methods are currently not available.

11.1.4 Determination of Appropriate Extraction Fluid

11.1.4.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

11.1.4.2 For wastes and wastewater, extraction fluid #1 should be used.

11.1.4.3 For cyanide-containing wastes and/or soil, extraction fluid #3 must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

11.1.4.4 If the aliquot of the sample used for the preliminary evaluation was determined to be 100% solid, then it can be used for the extraction (assuming at least 25 g remain). If the sample was subjected to the procedure in Step 11.1.1.7, then another aliquot must be used for the volatile extraction procedure. The amount of solid necessary depends on whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to nonvolatile extraction.

11.2 Procedure when Volatiles are not involved

A minimum sample size of 100 g (solid and liquid phases) is required. In some cases, a larger sample size may be appropriate depending on the solids content of the waste sample (percent solids); whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid; and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction so that the volume of the SPLP extract will be sufficient to support all the analytes required.

11.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, weigh out a subsample of the sample (100 g minimum) and proceed to Section 11.2.9.

11.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 6.0, Equipment and Supplies and is outlined in steps 11.2.3 to 11.2.8.

11.2.3 Pre-weigh the container that will receive the filtrate.

11.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals.

Note: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

11.2.5 Weigh out a subsample of the sample (100 g minimum) and record the weight on the leachate worksheet. If the waste contains < 0.5% dry solids,

the liquid portion of the waste, after filtration, is defined as the 1312 extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the 1312 extract. For wastes containing > 0.5% dry solids, use the percent solids information to determine the optimum sample size (100 g minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the 1312 extract.

11.2.6 Allow slurries to stand no longer than 2 hours so that the solid phase can settle. Samples may be centrifuged before filtration. Use centrifugation only as an aid to filtration. If the sample is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

11.2.7 Quantitatively transfer the sample (liquid and solid phases) to the filter holder. Spread the waste evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in section 11.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging; hence the need to slowly increase pressure.

11.2.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed or stored at 4°C until time of analysis.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

112.9 If the sample contains < 0.5% dry solids, and if particle-size reduction of the solid was needed, proceed to Section 11.2.13. If the sample contains > 0.5% dry solids, and if particle-size reduction of the solid was needed, proceed to Section 11.2.10. If the sample as-received passes through a 9.5-mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter paper used to separate the initial liquid from the solid phase, and proceed to Section 11.2.11.

11.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Section 11.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

Note: Sieving the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper or cloth) waste materials. Actual measurement of surface area in not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

11.2.11 Determine the amount of extraction fluid to add to the extractor. See section 12.0, Data Analysis and Calculation, for details.

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Close the extractor body tightly (use Teflon tape to ensure a tight seal), secure in rotary extractor device, and rotate at 30 rpm ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., room temperature at which extraction takes place) shall be maintained and at 23 ± 2°C during the extraction period. Record the temperature in the Synthetic Precipitation Leaching Procedure Worksheet and initial and date the entry.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g., limed or calcium carbonate-containing samples may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

11.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter. For final filtration of the 1312 extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) must be acid washed if evaluating the mobility of metals.

11.2.13 Prepare the 1312 extract as follows:

11.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained from Section 11.2.12 is defined as the 1312 extract. Proceed to Section 11.2.14.

11.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 11.2.12 with the initial liquid phase of the sample obtained in Section 11.2.7. This combined liquid is defined as the 1312 extract. Proceed to Section 11.2.14.

11.2.13.3 If the initial liquid phase of the waste, as obtained from Section 11.2.7, is not or may not be compatible with the filtered liquid resulting from Section 11.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the 1312 extract, and combine the results mathematically, as described in Section 11.2.14.

11.2.14 After collecting the 1312 extract, you must record the pH of the extract on the leachate worksheet. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid (HNO_3) to a pH <2. If precipitation is observed when you add nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses should not be acidified and the extract must be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until extraction is required. The 1312 extract is prepared and analyzed according to appropriate analytical methods. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and

combine the results mathematically by using a simple volume-weighted average. See Section 12.0, Data Analysis and Calculations, for details.

11.3 Procedure When Volatiles Are Involved

11.3.1 The ZHE device has approximately 500 ml internal capacity. The ZHE can accommodate a maximum of 25 grams of solid due to the requirement to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Charge the ZHE with sample only once and do not reopen the ZHE device. Always try not to expose the volatile sample to the atmosphere any more than is absolutely necessary.

11.3.2 When assembling the ZHE device, moisten the O-rings on the piston with extraction fluid. Adjust the piston so that the volume in the ZHE is just sufficient to hold the amount of sample being used. Secure the gas inlet/outlet flange onto the body and secure the glass fiber filter between the support screens and set aside. Set the inlet/outlet flange aside.

11.3.3 For samples that are 100% solid use the maximum 25 g for extraction. Record the weigh and proceed to Step 11.3.5.

11.3.4 Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For samples containing $\geq$0.5% dry solids, use the percent solids information obtained in 11.1.1 to determine the optimum sample size to charge into the ZHE. Weigh out a sub-sample of the appropriate size and record the weight.

Note: Some wastes, such as oil wastes and some paint wastes, will obviously contain some material that appears to be liquid. But even after applying pressure filtration this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

11.3.3.1 The recommended sample size is as follows:

- For samples containing <5% solids, weigh out a 500 gram sub-sample of waste and record the weight.
- For waste containing >5% solids, determine the amount of waste to charge into the ZHE as shown in Section 12.0, Data Analysis and Calculations.

11.3.5 If particle-size reduction of the solid portion of the sample was required in Step 11.1.3, proceed to Step 11.3.6. If particle size reduction was not required, proceed to Step 11.3.7.

11.3.6 Prepare the sample for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle size as described in Step 11.1.3.1. Waste and appropriate reduction equipment should be refrigerated to 4^{o} C prior to particle size reduction. Use mortar and pestle or other appropriate means to reduce particle size. The means used to effect PSR should not generate heat.

Note: Sieving of the waste is not recommended due to the possibility that volatiles may be lost.

11.3.7 Waste slurries do not have to stand to permit the solid phase to settle. Do not centrifuge, unless it can be done in the container that the sample came in originally.

11.3.8 Quickly quantitatively transfer the weighed sample (liquid and solid phase) to the ZHE, securing the support of the screens and top flange. Tighten all ZHE fittings and place the device in a vertical position (gas flange on bottom). Do not attach Tedlar bag.

Slowly apply pressure (1-10 psi) by connecting the nitrogen gas line to the bottom coupling to decrease headspace in the ZHE until liquid is observed from the top flange. Turn the pressure off and allow the waste to reach room temperature.

11.3.9 Attach the filter to the ZHE, and apply pressure. Gradually pressurize at 1-10 psi to force any liquid out of the ZHE and into the Tedlar bag. If no liquid appears or no more liquid has passed through the inlet/outlet valve on the top flange of the ZHE in a 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the inlet/outlet valve and no liquid has passed in a 2-minute interval, proceed to the next 10-psi increment. Stop filtration when the pressurizing gas begins to move through the valve or when liquid flow has ceased at 50 psi. This liquid should be saved and stored at 4°C until the leachate can be added to it.

Note: Applying too much pressure, or applying it too quickly can degrade the glass fiber filter and may cause premature plugging.

11.3.10 The material inside the ZHE is described as the solid phase of the sample and the filtrate is defined as the liquid phase. If the original waste contained <0.5% dry solids, this filtrate is defined as the extract ads is analyzed directly.

11.3.11 Determine the weight of extraction fluid (reagent water) #3 to put in the ZHE. Add the determined amount of extraction fluid to the ZHE through the inlet/outlet valve. Use the pressure can with the needle valve on the bottom open to allow the piston to move inside. Close the valves, disconnect the line from the pressure can, and manually agitate the ZHE device two or three times.

11.3.12 Add extraction fluid as follows.

11.3.12.1 Attach a line from the extraction fluid reservoir while the ZHE is in the vertical position. The line should be pre-flushed with fresh extraction fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston, open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Continue pumping fluid into the device until the appropriate amount has been introduced.

11.3.12.2 Close the valve, disconnect the extraction fluid line, and manually rotate end over end 2 or 3 times to ensure there is no leak. Pressurize the ZHE to 5-10 psi with the liquid inlet/outlet valve open to vent headspace into the hood. Immediately release the pressure when you see liquid, and close the inlet/outlet valve. Pressurize again at 5-10 psi to check that the ZHE fittings are secure, and that no leak is evident.

Secure the ZHE device in the rotary agitation device and rotate at 30 ± 2 rpm for 16-20 hours. Ambient temperature should be 23 ± 2°C during the extraction period.

11.3.13 After agitation is complete, check each ZHE to ensure that no leak has occurred by quickly opening and closing the inlet/outlet valve. If the ZHE device lost pressure, the sample must be extracted again.

Apply pressure (5-10 psi) to the ZHE and use a Tedlar bag to collect the leachate.

11.3.14 If the waste contained no initial liquid phase (filtrate), the liquid material obtained from the extraction is defined as the SPLP extract.

11.3.15 If compatible (i.e., will not form precipitate or multiple phases), the extracted filtered liquid phase of the waste as obtained in section 11.3.1 is combined with the leachate from 11.3.7.8. This combined liquid is defined as the SPLP extract.

11.3.16 If the initial liquid phase of the waste is not compatible with the extracted liquid resulting, then these liquids are not combined. These liquids are collectively defined as the SPLP extract, are analyzed separately, and the results are combined mathematically.

11.3.17 Determine the weight of the liquid phase by subtracting the weight of the filtrate container. The weight of the solid phase of the sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample. Record the weight of the liquid and solid phases.

11.3.18 Use whatever amount of solid is left in the ZHE extractor less than 25 g. If this does not support all necessary analyses, run multiple extracts of the sample and combine extracts before aliquoting.

11.3.18 For multiphasic samples, repeat step 11.3.3, except collect the leachate separately in another Tedlar bag. The initial fluid and the leachate should be analyzed separately and the results mathematically combined.

11.4 Preparation Blank

11.4.1 For each batch the preparation blank device should be made up of ZHE device pieces (barrel, piston, top flange, and base) etched with the same identification letter. All the pieces for the device used for the first batch should be assembled with the letter "A" on them. The pieces for the device used for the next batch will be lettered with a “B”. The assembled pieces should be used in alphabetical order. This is to ensure that the same device is not used for the preparation blank every time and that all devices are being cleaned after each batch. The ZHE devices used for samples can have mixed numbered pieces when assembled.

11.4.2 Once the ZHE device is assembled, follow the procedure for charging the ZHE device. Charge the ZHE with 500 ml of extraction fluid.

11.4.3 The prep blank should be collected and stored in the same manner as the waste samples until analysis.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculate the percent solids as follows:

$$Percent\ solids = \frac{weight\ of\ solid}{total\ weight\ of\ waste}\ x\ 100$$

12.2 Calculate the percent dry solids as follows:

$$Percent\ dry\ solids = \frac{(Weight\ of\ dry\ sample\ plus\ filter) - tared\ weight\ of\ filter}{Initial\ weight\ of\ sample}\ x\ 100$$

12.3 Determine the weight of waste to charge the ZHE as follows:

$$Weight\ of\ waste\ to\ charge\ ZHE = \frac{25}{percent\ solids}\ x\ 100$$

12.4 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$Weight\ of\ extraction\ fluid = \frac{20\ x\ \%solids\ x\ weight\ of\ waste\ filtered}{100}$$

12.5 If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average

$$Final\ analyte\ concentration = \frac{(V1)(C1) + (V2)(C2)}{V1 + V2}$$

where: V_1 = the volume of the first phase (L)
C_1 = the concentration of the analyte of concern in the first phase (mg/L)
V_2 = the volume of the second phase (L)
C_2 = the concentration of the analyte of concern in the second phase (mg/L)

13.0 Method Performance

Method performance is monitored through the use of sample duplicates at the rate of one in 20. Percent difference values are calculated.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, H_2SO_4, NaOH, ZN acetate are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW OLC02.1, OLC03.2, OLM04.2, **OLM04.3,** ILM04.1, ILM05.**2**, **ILM05.3,** plus revisions

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, "Synthetic Precipitation Leaching Procedure," Method 1312.

16.3 Methods for Chemical Analysis of Water and Wastes, March 1983

16.4 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.5 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.6 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.7 QCSOP: Proper Documentation Procedures

16.8 QCSOP: Numerical Data Reduction

16.9 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.10 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.11 NELAC Standards, **June 2000**, plus revisions

16.12 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.13 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.14 CompuChem Quality Manual, Revision **4**, 1**2**/**10**/0**4**, plus revisions

16.15 Sample Control SOP 4.1, "Receiving Samples"

16.16 Sample Control SOP 4.6, "Storing Samples"

16.17 QC SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.18 Sample Preparation Procedure SOP –157, "pH Measurement for Solid Samples by EPA CLP and NYSASP."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1--SPP-818 Synthetic Precipitation Leaching Procedure Worksheet

17.2 Attachment 2--Example of the TCLP Laboratory pH Meter Calibration Logbook

17.3 Attachment 3--Example of the TCLP Laboratory Balance Calibration Logbook

Attachment 1

CompuChem a division of Liberty Analytical Corp. Logbook 2 YY 1

Corning pH-30 Meter Calibration Log

Buffer 2.00	Buffer 4.00	Buffer 10.00	Date	Initials
If reading are not within ± 0.05 pH units of 2.00, 4.00, and 10.00, contact supervisor immediately. The pH meter cannot be used if readings are not within ± 0.05 pH units of the buffer solution value.				

Reviewed by: _____ Date: _____

Attachment 2

CompuChem a division of Liberty Analytical Corp. Logbook 2 PP 3

Top Loading Balance Calibration Log: Ohaus E400
Organic Prep. Lab

Balance serial no.: 1300851 Weight Set serial no.: 45824

Date	1.00 g (±0.03g)	10.00 g (±0.05g)	30.00 g (±0.07g)	50.00 g (±0.10g)	Initials	Comments

NOTE: Balance is tared with weighing vessel (i.e., weigh boat, beaker, etc.) prior to recording standard weight values.

Reviewed By: _____ Date: _____

3/25/02:dce

Attachment 3

COMPUCHEM

SW 846 METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE

ASSIGNED TO: _____ DATE EXTRACTED _____

EMPLOYEE ID _____ Non Volatile leaching _____ -818 Volatile leaching _____ Y/N

Y/N

SAMPLE #	EXT. FLUIDS (Vol. Added) 1	2	3	PARTICLE REDUCT. DONE (y/n)	FINAL LEACH pH VALUE	FINAL VOLUME (ml)	% Solids	COMMENTS
								ZHE'S CHECKED TO ENSURE
								PRESSURE MAINTAINED Y/ N
								ZHEBLK ZHE BODY # _____

LOADED TUMBLER CALIB. CHECK (MUST BE 30 RPM ρ 2 RPM) Room Temp _____

TUMBLER # | CALC. RPM

ROTATION TIME ONLY FINAL VOL. VERIFIED: _____ REVIEWED BY: _____

DATE/TIME STARTED _____/_____ DATE/TIME COMPLETED _____/_____

Ext. fluid 1 pH _____ Ext. fluid 2 pH _____ Ext. fluid 3 Reagent water

(4.20+ 0.05) (5.00 ρ 0.05) (Used for cyanide & VOA leaching)

{COUNT rpm FOR 30 sec. AND MULTIPLY NUMBER BY 2 TO CALCULATE RPM}

MANUFACTURER AND LOT NUMBER OF REAGENT USED: _____

Rev. 3/15/00.mli

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure __X__ outdated procedure (archive) _____

- Procedure Code: SPP-938 SOP Section #: 2.6.5 Revision #: 5

SOP Title: Automated Florisil Cartridge Clean-up for Pesticide/PCB Analysis by CLP and SW-846

Effective date: (QA fills in) 6-30-06

- Procedure prepared by: Bob McGee — Date: 6-29-06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) — Date: 6/29/06
- Reason for change: Revision requested by NJ DEP
- This procedure meets the requirements of the following approved method references: USEPA CLP SOW OLC03.2, OLM04.3, SOM01.1, plus revisions; SW-846, 3RD Edition, Update III, Method 3620B

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

_____ _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -938: Manual Florisil Cartridge Cleanup of Water and Soil Extracts for the Analysis of Pesticide/PCB by CLP **and** SW-846

Table of Contents

Sample Preparation Procedure -938: Automated Florisil Cartridge Cleanup for Pesticide/PCB Analysis by CLP **and** SW-846

1.0 Scope and Application

Florisil cartridge cleanup is required in the EPA Contract Laboratory Program (CLP) for all soil and water samples extracted for analysis of pesticides/Aroclors **in the OLM04.3 and OLC03.2 SOW documents and pesticides in the SOM01.1 SOW document**. Florisil cleanup is optional for SW-846. Florisil cleanup significantly reduces matrix interferences caused by polar compounds.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Method Summary

Extracts for Florisil cleanup **are** processed using a 10 mL syringe and Sep-pak cartridge. The analyst loads the syringe, and elutes sample extracts with 90:10 hexane:acetone. The pesticide extracts are then concentrated to appropriate final volumes and delivered to the GC laboratory with the necessary paperwork (Attachments 1 and 2) for analysis by GC/ECD.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this

minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

For CLP the reporting limit is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/L for waters and µg/kg for soils

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (17 calendar days if requested by the client) beginning with the receipt of the first sample.

3.5 VTSR – Validated Time of Sample Receipt, a CLP term

4.0 Interferences

Florisil cleanup significantly reduces matrix interferences caused by polar compounds.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are **required** to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Solvents – pesticide grade or equivalent

6.1.1 Hexane

6.1.2 Acetone

6.1.3 Hexane:acetone mixture (90:10) (v:v)

6.2 10 mL concentrator tube

6.3 Waters brand Sep-Pak Plus Florisil cartridge (1 gram)

6.3.1 Each lot is tested in the laboratory and approved before use.

6.4 **Vial – 10 mL**

7.0 Reagents & Standards

All standards are prepared **in** the Organic Standards **laboratory**. Details for the preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.) **Refrigerate the s**tandards in the laboratory at 2 – 4^{o} C**,** separately from samples**,** when not in use.

7.1 Reagent water – All water used **in this procedure must be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (19th and 20th Editions of Standard Methods, Method 1080), and is demonstrated to meet the blank criteria contained in this Standard Operating Procedure (SOP). It is referred throughout this SOP as DI water.**

7.2 Spike standard #4027 – 2,4,5-trichlorophenol solution (0.1 µg/mL in acetone)

7.3 GC midpoint calibration standard A (INDAM)

8.0 Sample Preservation **and** Storage

8.1 **Preserve and store s**amples according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 For CLP, **extract** aqueous samples within 5 days of VTSR and solid samples within 10 days. For SW-846, aqueous samples must be extracted within 7 days of sampling and soils within 14 days.

Note: Pesticide/PCB extracts are generated in the OLM04.3 and OLC3.2 SOW documents and require Florisil clean-up. In the SOM01.1 SOW document, only the pesticide extracts require Florisil clean-up.

8.3 **O**btain **samples** from the Custodian. **A**llow **samples** to come to room temperature prior to sample preparation. After preparation, return **samples** to the Custodian and placed in the cooler at 2 – 4^{o} C for long-term storage and disposal.

9.0 Quality Control

9.1 Florisil Cartridge Performance Check

9.1.1 Every lot number of Florisil cartridges must be tested by the following procedure and approved by the GC laboratory before it can be used for sample cleanup. **Conduct the performance check at least once on each Florisil lot, or every 6 months, whichever is more frequent.**

9.1.2 Waters Sep-pak plus cartridges are generally purchased in lots of 2000 each.

9.1.3 If cartridges do not meet QC requirements, return **them** to the vendor.

9.1.4 Prepare two lot tests (an original and a duplicate) by preparing each as follows:

9.1.4.1 Add 0.5 mL of spike standard **(#4025**) and 0.5 mL of GC midpoint Standard A (INDAM) **or Standard C for SOM01.1 (INDCM)** together in a **10 mL vial**. Dilute to **4**.0 mL with hexane. **Blow down to 0.5 mL. Dilute again to 2.0 mL with hexane.**

9.1.4.2 Attach labels to the culture tubes to designate the lot number and date run as shown in the table below.

Xxxxxxx	T5129-1
LOT TEST	LOT TEST
xx/xx/xx	07/15/95

The CCN will be designated as follows:

1, 2, etc. = replicate number
T5129 = Lot number

9.1.4.3 Perform the Florisil Cleanup Procedure in Section 11.0.

9.1.4.4 Reduce the final volume to 1.0 ml using the nitrogen blow down technique and submit to the GC laboratory for GC/ECD analysis.

9.1.4.5 Complete the Florisil Lot Test worksheet.

9.1.4.**6 Determine t**he recovery of each analyte for evaluation and reporting purposes.

9.1.4.**7** The lot of Florisil cartridges is acceptable if

9.1.4.**7**.1 all pesticides are recovered at 80-120%

9.1.4.**7**.2 the recovery of trichlorophenol is less than 5%

9.1.4.**7**.3 no peaks interfere with the target analytes are detected.

NOTE: If cartridges fail due to 2,4,5-trichlorophenol recovery, heat the cartridges to 100° C for ~ 1 hour, the repeat lot tests. If cartridges pass after heating, **heat** all cartridges in that lot before use.

9.2 **Use this clean-up procedure with a**ll associated QC samples.

10.0 Calibration & Standardization

NA

11.0 Procedure

Follow documentation requirements in Q**uality** C**ontrol** SOP **13.6 "**Proper Documentation Procedures**"**.

11.1 Florisil Cartridge Cleanup Procedure

11.1.1 For **OLM04.3 and SOM01.1** water samples, ensure that the hexane extract has been concentrated to 10 mL, then remove a 2.0 mL aliquot. **For OLC03.2 water extracts the hexane-exchanged extract is concentrated to 2.0 mL and the entire volume is subjected to Florisil clean-up.** For SW-846 soil samples not requiring GPC, after exchange to hexane, concentrate to 5.0 mL, then remove a 2.0 aliquot.

For CLP **(OLM04.3 and SOM01.1)** soil samples after GPC, concentrate the hexane-exchanged extract to 2.0 mL and florisil the entire extract.

11.1.2 Assemble a Florisil Sep-pak cartridge to a 10 mL syringe. Elute the cartridge with approximately 10-20 mL of 90:10 hexane:acetone, but do not let the cartridge go dry. Rinse with 2.0 mL hexane.

11.1.3 Transfer the 2.0 mL hexane extracts to the 10 mL syringe. Also, transfer labels with CompuChem number to concentrator tube.

Push the extract through, but do not allow the cartridge to go dry. Rinse the syringe with a small amount of hexane. Push this through, but do not allow to go dry.

Collect the eluant after Florisil into a concentrator tube. Next, elute with approximately 8 mL of 90:10 hexane:acetone. Collect this eluant into the same concentrator tube.

11.1.4 Concentrate to the final extract volume using the nitrogen blowdown technique or a micro Snyder. The final volume is 2.0 mL.

11.1.5 Complete the paperwork, post the extraction queues in LIMS, and label the extracts with CompuChem number, procedure code, and date. Sign the appropriate preparation worksheet to document the final volume was verified. Include on the worksheet, the manufacturer and lot number of reagents/solvents used.

11.1.6 Deliver the sample extracts with the completed paperwork to the designated area in the GC laboratory documenting custody transfer.

11.1.7 The extracts are now ready for analysis following **the appropriate instrumentation procedures.**

12.0 Data Analysis & Calculations

Calculations must be consistent with the Q**uality** C**ontrol** SOP **13.4 "**Numerical Data Reduction**"**.

13.0 Method Performance

This method was validated in conjunction with the analytical method through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data is retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has

established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. **See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.**

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW OLC03.2, OLM04.3, plus revisions**, SOM01.1 (May, 2005)**

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3620B

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.5 Q**uality** C**ontrol** SOP **13.6 "**Proper Documentation Procedures**"**

16.6 Q**uality** C**ontrol** SOP **13.4 "**Numerical Data Reduction**"**

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, July **2003**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, **December 2005**, plus revisions.

16.12 CompuChem Quality Manual, Revision **7**, **Update 1, 12/13/05**

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 - **Florisil Lot Test** Worksheet

Attachment 1

CompuChem a Division of Liberty Analytical

Worksheet Ref: Florisil Lot Test

EXTRACTION WORKSHEET

ASSIGNED TO: _____ **Pesticide/PCB** **DATE PREPPED** 5/22/2006

EMP. ID NUMBER _____ **FLORISIL LOT TEST** _____

	COMPUCHEM NUMBER	SAMPLE TYPE	FINAL VOLUME	COMMENTS
1		FLORISIL TEST SAMPLE	1.0ML	0.5 MLs OF 4025 AND MIX A MED 3/90
2		FLORISIL TEST SAMPLE	1.0ML	ARE DILUTED TO 4.0ML IN HEXANE, BLOWN DOWN TO 0.5ML
3		FLORISIL TEST SAMPLE	1.0ML	BROUGHT BACK UP TO 2.0ML W/ HEXANE, THEN FLORISILED
4		FLORISIL TEST SAMPLE	1.0ML	
5				
6				
7				
8				
9				
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25				
26				

4025 SOLUTION	AMT. 0.5ML LOT
MIX A-MED 3/90 SPIKE SOLUTION	AMT. 0.5ML LOT

FINAL VOLUME VERIFIED _____

SUPERVISOR REVIEWED _____

Rev. 5/11/06 jad

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-945 SOP Section #: 2.6.4 Revision #: 4.5 (Dr 2/24/04)

SOP Title: Sulfuric Acid Wash of PCB - Only Hexane Extract (SW-846)

Effective date: (QA fills in) 3/19/04

- Procedure prepared by: Diane C. Ellwanger Date: 2/25/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Linda Carter Date: 3/2/04
- Reason for change: OH VAP compliance
- This procedure meets the requirements of the following approved method references: SW-846, Update III, 3rd Edition, Method ~~8015B~~ (Dr 2/24/04) 3665A

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 2/10/05

Annual Review—Signature: _____ Date: 7/5/06

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -945: Sulfuric Acid Wash of PCB-Only Hexane Extract (SW-846)

Table of Contents

Sample Preparation Procedure -945: Sulfuric Acid Wash of PCB-Only Hexane Extract (SW-846)

1.0 Scope & Application

This sample preparation procedure is suitable for the cleanup of sample extracts prior to analysis for polychlorinated biphenyls. This method cannot be used to cleanup extracts for other target analytes, as it will destroy most organic chemicals including the pesticides Aldrin, Dieldrin, Endrin, Endosulfan (I and II), and Endosulfan sulfate. The method is based on SW-846 Method 3665A.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

2.1 The extract generated from the sonication procedure has already been exchanged to hexane. The entire extract is treated with concentrated sulfuric acid. Appropriate caution must be taken with the acid.

2.2 Blanks and other QC samples must be subjected to the same cleanup as the samples associated with them.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

3.3 An SDG is defined by the following, whichever is more frequent:

each 20 field samples received within a case, or

each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

Analytical range for organics – The analytical range is defined by the concentration of the highest standard in the initial calibration. Samples containing target analyte above this limit must be diluted, or a lesser amount of sample analyzed, in order to bring the concentration to within the upper half of the calibration range. For CLP the upper half is defined as the concentration between the midpoint standard and the high standard. For non-CLP the limit is between one-half the concentration of the high standard and the high standard. The analytical range for inorganics is defined by the quarterly linearity study.

4.0 Interferences

This technique will not destroy chlorinated benzenes, chlorinated naphthalenes (Halowaxes), and a number of chlorinated pesticides.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be

indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

Because concentrated acid is being employed, a full face shield must be worn.

6.0 Equipment & Supplies

6.1 Syringe or Class A volumetric pipet, glass; 1.0, 2.0 and 5.0 mL.

6.2 Vials - 1, 2 and 10 mL, glass with Teflon lined screw caps or crimp tops.

6.3 Kunderna-Danish (K-D) apparatus.

6.3.1 Concentrator tube - 10 mL graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

6.3.2 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

6.3.3 Springs - 1/2 inch (Kontes K-662750 or equivalent).

6.4 Vortex mixer.

6.5 Nitrogen evaporation device - equipped with a heated bath that can be maintained at 35°C, to 40°C, N-Evap by Organomation Associates, Inc. (or equivalent).

7.0 Reagents & Standards

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (18^{th} and 19^{th} Editions of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Sulfuric acid/Water, concentrated, reagent grade.

7.3 Hexane, C_6H_{14} - Pesticide grade or equivalent.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler at 2-4.4^{o} C for long-term storage and disposal. (Sample Preparation)

9.0 Quality Control

9.1 Contact the area supervisor is questions arise during this procedure.

9.2 All associated QC samples must also under this cleanup procedure.

10.0 Calibration & Standardization

Not applicable.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Sulfuric acid cleanup

Take the entire hexane extract and carefully add 2-5 mL of concentrated sulfuric acid.

Caution: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

11.1.2 Vortex or shake for approximately one minute.

Caution: Be extremely careful so no acid escapes.
AVOID SKIN CONTACT, SULFURIC ACID BURNS.

11.1.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

11.1.4 If a clean phase separation is achieved, proceed to 11.1.7.

11.1.5 If the hexane layer is colored or the emulsion persists for several minutes, remove the sulfuric acid layer from the vial and dispose of it properly. Add another 2-5 mL of concentrated sulfuric acid.

NOTE: Do not remove any hexane at this stage of the procedure.

11.1.6 Shake the sample for approximately one minute and allow the phases to separate.

11.1.7 Repeat steps 11.1.5 and 11.1.6 until the hexane layer is colorless or there is no visible change in successive sulfuric acid washes.

11.1.8 Transfer the hexane layer to a clean 10 mL vial. It is not critical to transfer the entire hexane layer. It is critical that no acid is transferred with the hexane since the acid will destroy a GC column.

11.2 Documentation

11.2.1 The extraction worksheet (Attachment 1) is completed by the technician performing the acid wash. The worksheet would have accompanied the samples prepared by the sonication extraction procedure, which then required the acid wash. Once completed, the worksheet accompanies the samples to the GC laboratory. Include on worksheet the manufacturers and lot numbers of reagent/solvent used.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or

eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, H_2SO_4, NaOH, ZN acetate are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, 3665A, (The modification uses concentrated sulfuric acid rather than 1:1 sulfuric acid/water and does not employ permanganate.)

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, June 2000, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **4**, 12/1**0**/0**3**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Extraction Worksheet

Attachment 1

CompuChem

ASSIGNED TO _____ EXTRACTION WORKSHEET DATE EXTRACTED/POSTED:

PCB ONLY IN S/S/S BY METHOD 3550A/3665A for 8082

EMPLOYEE ID # _____ -735 PCB's ONLY BATCH NO.:

	COMPUCHEM NUMBER	CLIENT SAMPLE ID	QC SAMPLE TYPE	SAMPLE WEIGHT (g)	FINAL VOLUME (ml)	COMMENTS
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						ACID WASH (3665A) 5 ML FINAL EXTRACT VOLUME
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						

		#	AMOUNT	LOT #	SURROGATE AND SPIKE ADDED BY:
SURROGATE		426	2.0 ml		_____ / _____ FINAL VOLUME VERIFI
SPIKE		4615	1.0 ml		INITIALS DATE

SUPERVISOR REVIEWEI

Analysts initials. Extract _____ KD _____ N2_____ Bottle up _____ Acid Wash _____ Witness _____/__

Initials

Note: Attachment is subject to change without notice.

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

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This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SAP-939 SOP Section #: 2.6.3 Revision #: 8

SOP Title: Gel Permeation Chromatography (GPC) Cleanup of Semivolatile Soil Sample Extracts by CLP, SW-846, and NYSASP

Effective date: (QA fills in) 3/19/04

- Procedure prepared by: Diane C. Ellmore Date: 2/25/04 or 3/25/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Linda Carter Date: 3/2/04
- Reason for change: OH VAP compliance
- This procedure meets the requirements of the following approved method references: US EPA CLP SOW OLM 04.3, plus revisions; SW-846, 3rd Edition, Update III, Method 3640A

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

ANNUAL

Annual Review—Signature: _____ Date: 2/10/05

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -939: Gel Permeation Chromatography (GPC) Cleanup of Semivolatile (SV) Soil Sample Extracts by CLP, SW846, and NYSASP

Table of Contents

Sample Preparation Procedure -939: Gel Permeation Chromatography (GPC) Cleanup of Semivolatile (SV) Soil Sample Extracts by CLP, SW846, and NYSASP

1.0 Scope and Application

GPC is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range of uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked styrene-divinyl benzene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

GPC is required for all soil/sediment samples, regardless of concentration level, for the elimination of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds from the sample extract. GPC is appropriate for both polar and non-polar analytes, therefore, it can be used effectively to clean up extracts containing a broad range of analytes.

Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue ultimately will reduce the chromatographic separation efficiency or column capacity because of absorption of the target analytes on the active sites. Pentachlorophenol especially is susceptible to this problem.

Method detection limit studies and reporting limits may be found in the Sample Preparation Procedures identified in the References, Section 16.0, of this SOP.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The semivolatile (SV) soil sample is extracted, concentrated, and filtered through a 0.5- or 0.45-micron Teflon membrane filter, diluted to 10.0 ml with methylene chloride, and

processed through GPC cleanup. High molecular weight interferences are removed based on "dump" and "collect" times, which are determined by using a GPC calibration standard containing corn oil (representing high molecular weight interferences), bis(2-ethylhexyl)phthalate (a SV compound), methoxychlor (a pesticide compound), perylene (a SV compound), and sulfur (optional) with the ultraviolet (UV) detector set at 254 nm. Dump and collect times are based on the elution times of the five compounds in the GPC calibration standard.

Five ml of the 10.0-ml sample extract are injected onto the GPC column. The high molecular weight interferences in the sample extract pass through the GPC column first and are eluted during the "dump" portion of the GPC run. The semi-volatile compounds of interest are then eluted to an Erlenmeyer flask during the "collect" portion of the GPC run. The GPC column is subsequently washed with methylene chloride for a minimum of 10 minutes before the next sample is loaded onto the GPC column. The collected sample extract is then concentrated to a final volume of 0.5 ml in methylene chloride and submitted to the GC/MS Laboratory for analysis following one of the Instrument Procedures listed in References, Section 16.0, of this SOP.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

For CLP the reporting limit is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/L for water and µg/kg for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SOP – Standard Operating Procedure

3.6 CRQL – Contract Required Quantitation Limit

3.7 Resolution - As defined in the CLP SOW, resolution (also termed separation or percent resolution) is the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100. The Glossary of Terms, Exhibit G-6, of the CLP SOW contains a diagram of peak resolution.

3.8 CLP SOW – Contract Laboratory Program Statement of Work

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2 Interferences may also be caused by carry-over from bis(2-ethylhexyl)phthalate and methoxychlor in the GPC calibration standard. Rigorous cleanup of the GPC instrument after each batch of samples is processed is performed by the GPC chemists. The cleanup may include backflushing the 24-position wafer valves inside the GPC unit, flushing the 23 uptake lines and solvent elution lines by

running methylene chloride blanks at each of the 23 positions, monitoring the uptake rate for all positions used for the last batch of samples run, and other maintenance as needed, including changing pump seals, changing the wafer valves, and packing new GPC columns.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 GPC cleanup device - GPC Autoprep Model 1002 A or B (Analytical Biochemical Laboratories, Inc., or equivalent). The system must meet the calibration requirements stated in Section 10.0 of this SOP. The system consists of the following components.

6.1.1 Chromatographic column - 700-mm X 25-mm I.D glass column.

6.1.1.1 Flow is upward. To simplify switching from the UV detector during calibration to the GPC collection device during extract cleanup, an optional double three-way valve (Rheodyne Type 50 Teflon Rotary Valve #10-262 or equivalent) may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

6.1.2 Guard column - (Optional) 5-cm, with appropriate fittings to connect to the inlet side of the GPC analytical column

6.1.2.1 Supelco 5-8319 or equivalent

6.1.3 Bio Beads (SX-3) - 200-400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, catalog 152-2750 or equivalent).

6.1.3.1 An additional 5 g of Bio Beads is required if the optional guard column is employed. The quality of the Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they also can pass through the column screens and damage the 24-position wafer valve.

6.1.4 Ultraviolet detector - fixed wavelength (254 nm) with semi-prep flow-through cell.

6.1.5 Strip-chart recorder, recording integrator, or laboratory data system.

6.1.6 Syringe - 10-ml with Luerlok fitting, Hamilton gas-tight syringe or equivalent.

6.1.7 Syringe filter assembly - disposable, (Gelman Sciences Acrodisc CR PTFE, Product No. 4219, 25-mm, 0.45-µm syringe filters, or equivalent).

6.1.7.1 Rinse each syringe filter with a minimum of 25 ml of methylene chloride prior to use.

7.0 Reagents

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remained of this SOP as DI water.

7.2 Solvents, pesticide grade or equivalent

7.2.1 Methylene chloride

7.2.2 butyl chloride

7.3 Calibration Standards

7.3.1 GPC Calibration solution contains the following in methylene chloride (in elution order):

Compound	Concentration (mg/ml)
corn oil	25.0
bis(2-ethylhexyl)phthalate	0.5
Methoxychlor	0.1
perylene	0.02
sulfur	0.08

7.3.1.1 Store the calibration solution in an amber glass bottle with a Teflon-lined screw-cap at 4° ± 2° C, and protect it from light. Refrigeration may cause the corn oil to precipitate. Therefore, before use, allow the calibration solution to stand at room temperature until the corn oil dissolves. Replace the calibration standard solution every 6 months, or more frequently, if the standard is shown to have degraded or concentrated.

7.3.2 The GPC Continuing Calibration Verification (CCV) solution is prepared the same as the GPC Calibration solution.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 A GPC reagent blank is analyzed weekly after the GPC calibration, or each GPC CCV, on each ABC GPC unit utilized for cleanup of semi-volatile samples.

9.1.1 The blank must not contain any target analytes above the CRQL.

9.1.2 If the GPC methylene chloride blank contains target compounds at or above the CRQL or phthalate esters at or above five times the CRQL, then the instrument must be further cleaned and another GPC method blank processed.

9.1.3 If the GPC methylene chloride blank fails the second time, the column may need to be changed. Check with the shift supervisor before changing the GPC column.

9.2 All associated QC samples must also under this cleanup procedure.

10.0 Calibration & Standardization

10.1 Calibration of the GPC Column

10.1.1 Verify the flow rate by collecting column eluate for 10 minutes in a graduated cylinder and measure the volume, which should be 45-55 ml (4.5-5.5 ml/minute). Once the flow rate is within the range of 4.5-5.5 ml/minute, record the room temperature and column pressure which should be between 6 and 10 psi.

10.1.2 Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not meet the criteria below would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads, if the column fails all the criteria.

10.1.2.1 The flow rate may need to be decreased when using the high pressure column. The normal pressure when using this column is approximately 200 psi.

10.1.3 While it is mandatory to run a weekly GPC calibration, it is optional to run one daily. Using a 10 mL syringe, load sample loop #1 with the GPC calibration solution. Record the lot number of the GPC calibration solution used on the appropriate GPC Weekly Calibration Log (see Attachment 1). With the ABC automated system, the 5-ml sample loop requires a minimum of 8 ml of the GPC calibration solution. Switch the outlet line from the GPC column to the UV detector inlet line.

10.1.3.1 A 2-ml injection loop may be used instead of the 5-ml loop. If used, the manufacturer's instructions should be followed.

10.1.4 Start the run program on the ABC GPC system with 60-minute dump time and obtain a UV trace showing discrete peaks for each of the components. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the requirements listed below.

10.1.4.1 Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the GPC calibration

solution to achieve results similar to those listed below. An analytical flow-through detector cell will require a much less concentrated solution than the semi-preparation cell and, therefore, the analytical cell is NOT acceptable for use.

10.1.4.2 UV Trace Acceptance Criteria

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

10.1.5 Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate the column eluate collection just before elution of bis(2-ethylhexyl)phthalate and after elution of corn oil. Stop the eluate collection shortly after the elution of perylene and before the elution of sulfur, if sulfur is contained in the GPC calibration standard. Use a wash time of 10 minutes after the elution of sulfur; use a wash time of 6 minutes after the elution of sulfur when using the high pressure column.

10.1.5.1 The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than ± 5.0 % between calibrations. If the retention time shift is >5.0 %, take corrective action related to the potential causes shown below.

10.1.5.2 Excessive retention time shifts are caused by the following:

- poor laboratory temperature control or system leaks
- an unstabilized column that requires pumping methylene chloride through it for several more hours or overnight

- excessive laboratory temperatures causing outgassing of the methylene chloride

10.1.6 Failure to meet the UV Trace and retention time acceptance criteria may result in the following corrective actions.

10.1.6.1 The column may be cleaned by processing several 5-ml volumes of butyl chloride throughout the system. Butyl chloride removes discoloration and particles that may have precipitated out of the methylene chloride extracts.

10.1.6.2 If a guard column is being used, replace it with a new one. This may correct the problem.

10.1.6.3 If column maintenance does not restore the performance of the column, the column must be repacked with new packing and recalibrated.

10.1.6.4 Record any corrective action taken for the GPC units, including the GPC columns, on the GPC Run/Maintenance Log (Attachment 2).

10.2 Preparation of the SV GPC blank

10.2.1 To prepare a SV GPC blank, fill a 5 mL GPC loop with 5 ml of methylene chloride using a syringe containing 8 mL of methylene chloride. Record all information, including the manufacturer and lot number of the methylene chloride used, on the GPC Weekly Methylene Chloride Blank for Semi-volatiles worksheet (see Attachment 3). Set the collect and dump times as calculated from the UV trace.

10.2.2 Concentrate the collected GPC eluate using a Kuderna-Danish (K-D) evaporative flask, three-ball macro Snyder column, and 10.0-ml graduated concentrator tube, to a final volume of 0.5 ml and submit to GC/MS for analysis following Instrument Procedure 484, "Analysis of Extractable Semivolatiles in Aqueous and Solid Sample Extracts by EPA CLP and NYSASP".

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The GPC chemist is responsible for accurately filling in the worksheet in all

the applicable columns. If any of the four rows, one for each of the four ABC GPC units, are not used, the GPC chemist draws a line through that particular row or rows. A copy of the UV trace of the calibration solution must be submitted along with the completed appropriate Extraction Worksheet (Attachment 4 shows Procedure –738 worksheet). Samples are extracted prior to GPC cleanup following one of the Sample Preparation Procedures listed in the References, Section 16.0, of this SOP.

11.1 Column Preparation

11.1.1 Weigh out 70 g of Bio Beads (SX-3) in a 500-ml beaker and add approximately 300 ml of methylene chloride. Swirl the container to ensure wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times. If a guard column is to be used, repeat the above steps with 5 g of Bio Beads in a 125-ml bottle or beaker, using 25 ml of methylene chloride.

11.1.2 Turn the column upside down from its normal position, and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near the end as possible, but no closer than 5 cm (measured from the gel packing to the collar).

11.1.3 Raise the end of the outlet tube to keep the solvent in the GPC column. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

11.1.4 Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500-ml separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column, and allow the excess solvent to drain. Raise the tube to stop the flow when the top of the gel begins to look dry. Add additional methylene chloride to just rewet the gel.

11.1.5 Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the

seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.1.6 Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column.

Repeat the step in paragraph 11.1.5 and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

11.1.7 Push the plunger until it meets the gel; then, compress the column bed about four centimeters.

11.1.8 Pack the optional 5-cm guard column with approximately 5 g of pre-swelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

11.1.9 Connect the column inlet to the solvent reservoir (the reservoir should be placed higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1/16" OD x 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 ml/min for 1 hr.

11.1.10 After washing the column for at least 1 hour, connect the column outlet tube to the inlet side of the UV detector. A restrictor (same size as the one in paragraph 11.1.9) in the outlet tube from the UV detector will prevent bubble formation, which causes a noisy UV baseline. The restrictor will not affect flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi backpressure is achieved.

Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.1.11 When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.1.12 An alternative column would be a pre-packed stainless steel high pressure column. Typical pressure on this column would be approximately 200 psi.

11.2 The system is now ready for calibration. See Section 10.0 above for the procedure and further details of calibration.

11.2.1 Calibrate the GPC upon contract award and run the calibration verification at least once every seven days of sample cleanup. The UV trace must meet requirements and the retention times of the calibration compounds must be within 5.0% of their retention times in the previous calibration.

11.2.2 A copy of the two most recent UV traces of the calibration solution must be submitted along with the completed extraction worksheet.

11.3 Sample Extract Cleanup

11.3.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift, and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 24° C.

11.3.2 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution must be diluted and loaded into several positions. Similarly, extracts containing more than 40 mg/ml of nonvolatile residue must be diluted and loaded onto several positions. The nonvolatile residue may be determined by evaporating a

100-µl aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

Warning: Do not load extremely dirty samples on the high pressure column. If the column is damaged, it will have to be replaced.

11.3.3 Particles greater than 0.5 microns may scratch the wafer valves, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 0.5-micron Teflon filter disc by attaching a syringe filter assembly containing the filter disc to a 10-ml syringe. Prep (rinse) the filter body with 25-30 ml of methylene chloride prior to use. Remove the plunger from a 10-ml glass syringe and pour the 10.0-ml sample extract into the glass syringe body with the 0.5-micron Teflon filter already attached to the syringe body. Replace the plunger into the syringe body and gently depress the plunger to filter the sample extract through the filter. The filtered sample is placed in either a 20-ml Class A vial and capped with an aluminum seal with a Teflon-faced septum, or filtered directly into an ABC GPC tube. A volume of 7.5 mL of extract is loaded into a GPC tube, leaving 2.5 mL for back-up.

Note: While transferring the samples from the culture tubes to the GPC tubes, be sure that the GPC tube has a label specifying the CCN for the sample being loaded in that tube. After loading the sample, transfer the label on the culture tube to the receiving flask to complete the sample loading. THEN continue to the next sample.

11.3.4 Collect each sample extract in a 250-ml Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation. Monitor sample volumes collected.

11.3.4.1 The volume of GPC eluate collected for each sample extract processed through GPC cleanup may be used to indicate problems with the system during sample processing. The GPC chemist will note any collected volume variations in the GPC Run/Maintenance Log (Attachment 2) as well as any corrective action taken.

11.3.4.2 Changes in sample volumes collected may indicate one or more of the following problems.

- Change in solvent flow rate caused by channeling in the column or changes in column pressure.

- Increase in column operating pressure due to the adsorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

- Leaks in the system or significant variances in room temperature.

11.4 Final Concentration of Extract

11.4.1 Transfer the sample extract to a K-D flask after attaching a 10-ml graduated concentrator tube to the bottom of the K-D with rubber bands and/or blue Keck clips. Add 1-2 silicon boiling chips to the K-D flask, then attach a three-ball macro Snyder column to the top of the K-D flask. Pre-wet the Snyder column with 2-4 ml of methylene chloride and place the K-D apparatus on a water bath with the temperature of the water bath set between 80-90° C. The balls in the Snyder column will actively chatter as the methylene chloride evaporates. The extract should concentrate in 10-15 minutes, if not, adjust the water temperature as required. Remove the extract from the water bath when the apparent volume reaches 4 ml. Allow to cool and drain for at least 10 min. Remove the Snyder column, then remove the concentrator tube. Alternatively, microsnyder concentration may be used.

11.4.2 To complete the final extract concentration, place the concentrator tube onto an N-EVAP from Organomation Associates, Inc. The N-EVAP uses nitrogen to further concentrate the solvent extract. Concentrate the extract using a gentle stream of clean, dry nitrogen. Rinse the walls of the concentrator tube several times with methylene chloride during the final concentration.

CAUTION: The extract must never be allowed to become dry.

11.4.3 Quantitatively, concentrate the extract to a final volume of 0.5 ml and bottle in an amber GC autosampler vial, with screw-cap and Teflon-faced septum.

Note: Any samples that were loaded into two or more loops must be recombined before concentrating.

11.4.4 Using an orange label for semi-volatile samples, place the following information on the label with a black permanent labeling pen:

CompuChem number	XXXXXX
Procedure Code	-738 or –739
Extractoin date	XX/XX/XX

11.4.5 The extracts are now ready for GC/MS analysis following one of the Instrument Procedures listed in References, Section 16.0, of this SOP. Deliver the extracts to the designated area in the SV GC/MS instrument laboratory with all associated paperwork.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated in conjunction with analytical methods through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW for Organic Analysis, Multi-Media, Multi-Concentration OLM04.2, OLM04.3, plus revisions

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Method 3640A

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.5 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.6 QCSOP: Proper Documentation Procedures

16.7 QCSOP: Numerical Data Reduction

16.8 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.9 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.10 NELAC Standards, June 2000, plus revisions

16.11 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.12 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.13 CompuChem Quality Manual, Revision **4**, 12/**10**/**03**, plus revisions

16.14 Sample Control SOP 4.1, "Receiving Samples"

16.15 Sample Control SOP 4.6, "Storing Samples"

16.16 Instrument Procedure 484, "Analysis of Extractable Semivolatiles in Aqueous and Solid Sample Extracts by EPA CLP and NYSASP"

16.16 Sample Preparation Procedure –1015, "Preparation of Water Samples for the Analysis of Semivolatiles by CLP and NYSASP"

16.17 Sample Preparation Procedure –725, "Preparation of S/S/S Samples for Analysis of Low Level Semivolatiles by CLP and NYSASP"

16.18 Sample Preparation Procedure –1081, "Preparation of Water Samples for the Analysis of Semivolatiles by EPA CLP and NYSASP"

16.19 Sample Preparation Procedure –738, Sample Preparation of S/S/S for the Analysis of Low Level Semvolatiles by CLP and NYSASP"

16.20 Sample Preparation Procedure –079, "Preparation of Water Samples for the Analysis of Low Level Semivolatiles by SW846 and NYSASP"

16.21 Sample Preparation Procedure –176, "Preparation of S/S/S Samples for the Analysis of Low Level Semivoatiles by SW864 and NYSASP"

16.22 Instrument Procedure 463, "Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by EPA CLP and NYSASP"

16.23 Instrument Procedure 477, "Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW846 and NYSASP"

17.0 Documentation Description/Examples

17.1 Attachment 1 - GPC Weekly Calibration Log

17.2 Attachment 2 - GPC Run/Maintenance Log

17.3 Attachment 3 - GPC Weekly Methylene Chloride Blank for Semivolatiles worksheet.

17.4 Attachment 4 – Extraction Worksheet (SPP-738)

Attachment 1

COMPUCHEM a division of Liberty Analytical CORP. LOGBOOK 2 R 11

GPC Weekly Calibration Log: ABC #4

Tape a copy of the U/V trace in the space below. Make copies of this page and attach them to the associated extraction sheets. Label each peak on the U/V trace with the compound name. Show resolution calculations.

Date/Time: _____ Room Temp: _____ Technician Name/ID: _____

GPC calibration standards (listed in order of elution)	**Time** (min) Or (CM)	**RSD** (%)
Corn oil		
Bis(2-ethylhexyl)phthalate	_____	_____
Methoxychlor		
Perylene	_____	_____
Sulfur		

Lot No. of GPC Calibration Standard Used _____

Acceptance criteria: ± 5.0% change from previous calibration (Do not round.)
Retention time criteria met. Y N

Calculate and record retention time shift from the previous calibration/UV trace.

Flow Rate: 5.0 ml/min Column: Bio-Beads Chart Speed: 15 cm/hr

Column Pressure: _____psi

Pest/PCB		**Semivolatiles**
Dump Time: _____min	> 85% resolution between corn oil and phthalate Y N	Dump Time: _____min
Collect Time: _____min	> 85% resolution between phthalate and methoxychlor Y N	Collect Time: _____min
Collect Volume: _____ml	> 90% resolution between perylene and sulfur Y N	Collect Volume: _____ml
Wash Time: 10:00 min		Wash Time: _____ min

Reviewed By: _____ **Date:** _____

4/7/00:mlj

Note: Attachment is subject to change without notice.

Attachment 2

ABC GPC #4 Run/Maintenance Log

CompuChem a division of Liberty Analytical Corp. LOGBOOK 2 V 6

GPC Run Date: _____ **Analyst:** _____

Position	Sample ID	Position	Sample ID
1		13	
2		14	
3		15	
4		16	
5		17	
6		18	
7		19	
8		20	
9		21	
10		22	
11		23	
12			

Dump Time:_____

Collect Time: _____

Wash Time: _____

Head Pressure: _____

o GCP tubes cleaned prior to use
o Position cleaned prior to use
o Viewed first sample uptake
oFilled in GPC data on worksheet

Date of corn oil calibration _____

For Pests, date of weekly standards _____

For SV, date of GPC blanks _____

Comments/observations _____

_____

_____

_____

_____

Maintenance performed, if any

Reviewed By: _____

Date: _____

6/29/01:dce

Note: Attachment is subject to change without notice.

Attachment 3

COMPUCHEM a division of Liberty Analytical Corp.

SEMI-VOLATILE GPC WEEKLY METHYLENE CHLORIDE BLANK
USEPA CLP SOW
-939

TECHNICIAN NAME: _____ DATE RAN: _____

EMPLOYEE ID: _____ GPC INSTRUMENT #: **ABC GPC #** _____

SAMPLE ID NUMBER	GPC INJ. VOLUME	FINAL VOLUME	COMMENTS
A B	5.0 ml	0.5 ml	METHYLENE CHLORIDE BLANK
			MANUFACTURER & LOT #

* COMPLETE SAMPLE ID NUMBER USING GPC COLUMN NUMBER ALONG WITH MONTH AND DATE, (i.e. A10202B)

RUN WEEKLY GPC CALIBRATION THROUGH GPC CLEANUP EVERY SEVEN CALENDAR DAYS AND SUBMIT TO GC/MS FOR ANALYSIS.

A_____B = METHYLENE CHLORIDE ONLY.

SUPERVISOR REVIEW: _____

FINAL VOLUME VERIFIED: _____

EXTRACTS RECEIVED BY: _____

6/20/01:dce

Note: Attachment is subject to change without notice.

Attachment 4

EXTRACTION WORKSHEET
Semi-volatile Low. Level Soil EPA CLP SOW OLM 4.2

ASSIGNED TO _____ EMPLOYEE ID # _____ DATE EXTRACTED/POSTED _____

	Sample Number	Case #	QC SAMPLES TYPE	Sample Weight (g)	Final Volume (ml)	-738 GPC Final Vol (ml)	COMMENTS
1					10.0		
2					10.0		
3					10.0		
4					10.0		
5					10.0		
6					10.0		
7					10.0		
8					10.0		
9					10.0		
10					10.0		
11					10.0		
12					10.0		
13					10.0		
14					10.0		
15					10.0		
16					10.0		
17					10.0		
18					10.0		
19					10.0		
20					10.0		
21					10.0		
22					10.0		
23			BLK		10.0		
24			LCS		10.0		

GPC Instr. #_____

GPC Calib Date A_____
GPC Run Date_____

FINAL VOLUME VERIFIED: _____

REVIEWED BY:

	ID#	AMT	LOT #	SURR. & SPIKE ADDED BY
SURROGATE	431	0.5 ml		_____/_____
SPIKE	8003	0.5 ml		INITIALS DATE
Date GPC MeC12 Blank Done				Witness _____/_____ Initials Date

Analysts initials. Extracted _____ KD _____ N2_____ Bottle up _____

MANUFACTURER AND LOT NO. OF REAGENTS/SOLVENTS USED_____ 5/22/01:dce

Note: Attachment is subject to change without notice.

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-940 SOP Section #: 2.6.1 Revision #: 9

SOP Title: Gel Permeation Chromatography (GPC) Clean-up of Soil and Water for GC/ECD Analysis of Pesticides/PCBs by EPA CLP

Effective date: (QA fills in) 7/11/06

- Procedure prepared by: Bob Moran Date: 07-11-2006
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 7/11/06
- Reason for change: To clarify the concentration of single component pesticides injected onto the 5 mL GPC injection loop.
- This procedure meets the requirements of the following approved method references: US EPA CLP SOW OLM04.3 and SOM01.1, plus revising SW-846 3rd Edition; Update III, Method 3640A

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -940: Gel Permeation Chromatography (GPC) Cleanup of Soil and Water Extracts for GC/ECD Analysis of Pesticides/PCBs by EPA CLP

Table of Contents

Sample Preparation Procedure -940: Gel Permeation Chromatography (GPC) Cleanup of Soil and Water Extracts for GC/ECD Analysis of Pesticides/PCBs by EPA CLP

1.0 Scope and Application

GPC is a size exclusion technique that may be used to remove high molecular weight compounds from sample extracts. The removal of such compounds from sample extracts is desirable because they may contaminate injection ports, cause interferences, and accelerate column degradation.

GPC cleanup is required for all pesticide/PCB soil sample extracts under the OLM04.3 SOW and must be performed on all water sample extracts that contain higher molecular weight contaminants that interfere with the analysis of target compounds. It is a mandatory clean-up technique for pesticide extracts under the SOM01.1 SOW and is an optional clean-up technique for Aroclor extracts under that SOW, optional depending on the client submitting the samples.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Sample extracts are prepared in methylene chloride, filtered, and then injected into a GPC unit. A single fraction is collected for each extract. The collection time interval is determined by a calibration standard. All eluant before and after the collection time is diverted to waste. This cleanup procedure collects all analytes of interest and removes high molecular weight compounds which may adversely affect column performance and longevity.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

For CLP the reporting limit is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/L for water, µg/kg for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

3.5 SOP – Standard Operating Procedure

3.6 CLP SOW – Contract Laboratory Program Statement of Work

3.7 Resolution – As defined in the CLP SOW, resolution (also termed separation or percent resolution) is the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100. The Glossary of Terms, Exhibit G-6, of the CLP SOW contains a diagram of peak resolution.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2 Interferences may also be caused by carry-over from bis(2-ethylhexyl)phthalate and methoxychlor in the GPC calibration standard. Rigorous cleanup of the GPC instrument after each batch of samples is processed is performed by the GPC chemists. Any or all of the following steps can be taken to eliminate problems: back flushing the 24-position wafer valves inside the GPC unit; flushing the 23 uptake lines and solvent elution lines by running methylene chloride blanks at each of the 23 positions; and other maintenance as needed, including changing packing new GPC columns.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 GPC cleanup device – GPC Autoprep Model 1002A or 1002B (Analytical Biochemical Laboratories, Inc., or equivalent). The system must meet the calibration requirements stated in Section 10.0 of this SOP. The system consists of the following components.

6.1.1 Chromatographic column – 700 mm X 25 mm I.D glass column.

6.1.1.1 The flow through the column is upward.

6.1.2 Bio Beads (SX-3) – 200 - 400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, catalog 152-2750 or equivalent).

6.1.2.1 An additional 5 g of Bio Beads is required if the optional guard column is employed. The quality of the Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they also can pass through the column screens and damage the 24-position wafer valve.

6.1.3 Ultraviolet detector – fixed wavelength at 254 nm with semi-prep flow-through cell, Linear, Model 0106-0000.

6.1.4 Strip-chart recorder, Instruments Corp., Linear 1100

6.1.5 Syringe – 10 mL with Luerlok fitting, Hamilton gas-tight syringe or equivalent.

6.1.6 Syringe filter assembly – disposable, Gelman Sciences Acrodisc CR PTFE, Product No. 4219, 25 mm, 0.45 µm syringe filters, or equivalent

6.1.6.1 Rinse each syringe filter with a minimum of 25 mL of methylene chloride prior to use.

7.0 Reagents

All standards are prepared in the Organic Standards Laboratory. Details for the preparation are contained in the standard operating procedures (SOP) for that area. Standards are stored separately from samples at 2-4.4^{o} C in the laboratory when not in use.

7.1 Reagent Water-All water used in this procedure must be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (19th and 20th Editions of Standard Methods, Method 1080), and is demonstrated to meet the blank criteria contained in this Standard Operating Procedure (SOP). It is referred throughout this SOP as DI water.

7.2 Solvents - pesticide grade or equivalent

7.2.1 Methylene chloride, pesticide grade

7.2.2 Butyl chloride, pesticide grade

7.3 Calibration Standards

7.3.1 The GPC calibration solution in methylene chloride contains the following analytes (in elution order):

Compound	Concentration (mg/mL)
Corn oil	25.0
Bis(2-ethylhexyl)phthalate	0.50

Compound	Concentration (mg/mL)
Methoxychlor	0.10
Perylene	0.020
Sulfur	0.080

7.3.1.1 Store the calibration solution in an amber glass bottle with a Teflon®-lined screw cap at 4° ± 2° C, and protect it from light. Refrigeration may cause the corn oil to precipitate. Therefore, before use, allow the calibration solution to stand at room temperature until the corn oil dissolves. Replace the calibration standard solution every 6 months, or more frequently, if the standard is shown to have degraded or concentrated.

7.3.2 **When injected onto a 5 mL GPC injection loop, the single component compounds are present at the concentrations shown in the following table (for the two SOW documents).**

Compound	OLM04.3 SOW	SOM01.1 SOW
	Concentration (µg/mL)	Concentration (µg/mL)
gamma-BHC (lindane)	0.10	0.02
Heptachlor	0.10	0.02
Aldrin	0.10	0.02
4,4′-DDT	0.20	0.04
Endrin	0.20	0.04
Dieldrin	0.20	0.04

7.3.2.1 The solution for the OLM04.3 SOW is prepared by taking 4 mL of standard #4033 and diluting it to 10 mL. Standard #4033 contains lindane, heptachlor, and aldrin at 0.25 µg/mL and 4,4′-DDT, endrin, and dieldrin at 0.5 µg/mL. For the SOM01.1 SOW, 800 µL of standard #4033 is taken and diluted to 10 mL. (The standard is purchased from Restek as an ampulated certified solution.)

7.3.2.2 The Aroclor mixture contains 2 µg/mL each of Aroclor 1016 and 1260.

Note: The Aroclor mixture is used only for the OLM04.3 SOW.

7.3.2.3 The GPC calibration verification solution is stored in amber glass bottles with Teflon®-lined screw cap at 4°C (± 2°C). Replace the solution after six months or sooner if degradation/evaporation occur.

8.0 Sample Preservation and Storage

Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 A GPC reagent blank is generated weekly after the GPC calibration.

9.1.1 The blank must not contain any target analytes above the CRQL.

9.2 All associated QC samples must also undergo this cleanup procedure.

10.0 Calibration & Standardization

10.1 Calibration of the GPC Column

10.1.1 Verify the flow rate by collecting the column eluate for 10 minutes in a graduated cylinder. Measure the volume, which should be between 45 - 55 mL (4.5 - 5.5 mL/minute). Once the flow rate is within the range of 4.5-5.5 mL/minute, record the room temperature and the column pressure, which should be between 6 and 10 psi.

Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not meet the criteria below would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads, if the column fails all the criteria.

10.1.1.1 Flow rate may have to be decreased on the high-pressure column. The normal pressure when using this column is approximately 200 psi.

10.1.2 Using a 10 mL syringe, load sample loop #1 with the GPC calibration solution. Record the lot number of the GPC calibration solution used on the GPC Weekly Calibration Log (Attachment 1). With the ABC

automated system, the 5 mL sample loop requires a minimum of 8 mL of the GPC calibration solution. Switch the outlet line from the GPC column to the UV detector inlet line.

NOTE: A 2 mL injection loop may be used instead of the 5 mL loop. If used, the manufacturer's instructions should be followed.

10.1.3 Start the run on the ABC GPC system with a 60-minute dump time programmed in, and obtain a UV trace showing discrete peaks for each of the components. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the requirements stated below. Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the GPC calibration solution to achieve similar results. An analytical flow-through detector cell will require a much less concentrated solution than the semi-prep cell and, therefore, the analytical cell is NOT acceptable for use.

10.1.3.1 UV Trace Acceptance Criteria

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

10.1.4 Determine the elution times for the phthalate, methoxychlor, and perylene peaks. Phthalate will elute first, perylene last. Choose a "DUMP" time which removes > 85% of the phthalate. Choose a "COLLECT" time that will allow > 95% of the methoxychlor to be collected, and continue to collect until just prior to the elution of sulfur. Use a wash time of 10 minutes; use a wash time of 6 minutes on the high pressure column.

10.1.4.1 The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than ± 5.0% between calibrations. If the

retention time shift is >5.0%, take corrective action related to the potential causes shown below.

10.1.4.2 Excessive retention time shifts are caused by the following:

- poor laboratory temperature control or system leaks
- an unstabilized column that requires pumping methylene chloride through it for several more hours or overnight
- excessive laboratory temperatures causing outgassing of the methylene chloride

10.1.5 Failure to meet the UV Trace and retention time acceptance criteria results in one of the following corrective actions.

10.1.5.1 The column is cleaned by processing several 5 mL volumes of butyl chloride throughout the system. Butyl chloride removes discoloration and particles that may have precipitated out of the methylene chloride extracts.

Note: Backflushing with 2 mL aliquots of toluene is an acceptable substitute.

10.1.5.2 If a guard column is being used, replace it with a new one. This may correct the problem.

10.1.5.3 If column maintenance does not restore the performance of the column, the column must be repacked with new packing, or replaced, and recalibrated.

10.1.5.4 Record any corrective action taken for the GPC units, including the GPC columns, on the GPC Run/Maintenance Log.

10.1.6 Preparation of the GPC Blank

10.1.6.1 A GPC blank is generated weekly with the GPC calibration.

10.1.6.2 The GPC blank is processed by loading 5 mL of methylene chloride into a sample loop, processing it through the GPC unit, collecting it, exchanging it into hexane, and concentrating it to 5.0 mL final volume. Record all information,

including the manufacturer and lot number of the methylene chloride used, on the Pest/PCB Weekly GPC Calibration Check worksheet (Attachment 2). Set the collect and dump times as calculated from the UV trace.

10.1.6.1 Concentrate the collected GPC eluate using a Kuderna-Danish (K-D) evaporative flask, three-ball macro Snyder column, and 10.0 mL graduated concentrator tube, to a final volume of 0.5 mL and submit to GC for analysis following Instrument Procedure 181, "GC/ECD Analysis of Pesticides/PCBs in Aqueous and Solid Samples in the EPA CLP and NYSASP".

10.2.7 GPC Calibration Verification

10.2.7.1 Every seven calendar days the GPC system must be checked using the calibration verification solutions described in Section 7.0 above. The GPC calibration verification must be performed immediately following the GPC calibration. Separate 5 mL loops are loaded with these two solutions by using a 10 mL syringe containing 8 mL of each solution.

10.2.7.2 These calibration solutions are run through the system and collected using the time sequences established by the calibration procedures. The separate fractions are exchanged to hexane, and adjusted to a final volume of 10 mL. They are then submitted to the GC lab for analysis. The lot numbers of the GPC calibration verification (#4033), and the Aroclor 1016/1260 mixture **(for OLM04.3 only)** must be recorded on the Pest/PCB Weekly GPC Calibration Check worksheet.

10.2.7.3 The pattern of the Aroclor peaks **(for OLM04.3 only)** and analyte recovery must be determined for evaluation and reporting purposes.

- Analyte recovery must be within 80 - 120% of target
- The Aroclor elution pattern **(for OLM04.3 only)** must be the same as with previously run standards.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. A copy of the UV trace of the calibration solution must be submitted along

with the completed appropriate Extraction Worksheet (Attachment 3 shows Procedure – 726 worksheet). Samples are extracted prior to GPC cleanup following one of the Sample Preparation Procedures listed in the References, Section 16.0, of this SOP.

Note: It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift, and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 24° C.

11.1 Column Preparation

11.1.1 Weigh out 70 g of Bio Beads (SX-3) in a 500 mL beaker and add approximately 300 mL of methylene chloride. Swirl the container to ensure wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times. If a guard column is to be used, repeat the above steps with 5 g of Bio Beads in a 125 mL bottle or beaker, using 25 mL of methylene chloride.

11.1.2 Turn the column upside down from its normal position, and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near the end as possible, but no closer than 5 cm (measured from the gel packing to the collar).

11.1.3 Raise the end of the outlet tube to keep the solvent in the GPC column. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

11.1.4 Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500 mL separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column, and allow the excess solvent to drain. Raise the tube to stop the flow when the top of the gel begins to look dry. Add additional methylene chloride to just rewet the gel.

11.1.5 Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.1.6 Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column.

Repeat step 11.1.5 and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

11.1.7 Push the plunger until it meets the gel; then, compress the column bed about four centimeters.

11.1.8 Pack the optional 5-cm guard column with approximately 5 g of pre-swelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

11.1.9 Connect the column inlet to the solvent reservoir (the reservoir should be placed higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1/16" OD x 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 mL/minute for 1 hour.

11.1.10 After washing the column for at least 1 hour, connect the column outlet tube to the inlet side of the UV detector. A restrictor (of the same size as described in 11.1.9) in the outlet tube from the UV detector will prevent bubble formation, which causes a noisy UV baseline. The restrictor will not affect flow rate. After pumping methylene chloride through the column for an additional 1 - 2 hours, adjust the inlet bed

support plunger until approximately 6 - 10 psi backpressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.1.11 When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.1.12 An alternative column would be a pre-packed high pressure stainless steel column and guard column. Typical pressure on this column would be approximately 200 psi.

11.2 The system is now ready for calibration. See Section 10.0 above for the procedure and further details of calibration.

11.2.1 Calibrate the GPC upon contract award and then at least once per week. A calibration verification standard must also be run at least once per seven days, immediately following the GPC calibration. The UV trace must meet requirements and the retention times of the calibration compounds must be within 5.0% of their retention times in the previous calibration.

11.3 Sample Extract Cleanup

11.3.1 The 10 mL extract is delivered to the GPC station. The GPC chemist aliquots 4.0 mL, dilutes to 10 mL with methylene chloride and uses that for GPC processing.

In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol: water solution must be diluted and loaded into several positions. Similarly, extracts containing more than 40 mg/mL of nonvolatile residue must be diluted and loaded onto several positions. The nonvolatile residue may be determined by evaporating a 100-µl aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

NOTE: Do not run extremely dirty samples on the high-pressure column. If the column is damaged, it will have to be replaced.

11.3.2 Particles greater than 0.5 microns may scratch the wafer valves, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 0.5-micron Teflon® filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Prep (rinse) the filter body with 25 - 30 mL of methylene chloride prior to use. Remove the plunger from a 10 mL glass syringe and pour the 10.0 mL sample extract into the glass syringe body with the 0.5 micron Teflon® filter already attached to the syringe body. Replace the plunger into the syringe body and gently depress the plunger to filter the sample extract through the filter. The filtered sample is placed in either a 20 mL Class A vial and capped with an aluminum seal with a Teflon®-faced septum, or filtered directly into an ABC GPC tube. A minimum of 8.0 mL of filtered extract is required for the ABC GPC system to process each sample.

Note: While transferring the samples from the culture tubes to the GPC tubes, be sure that the GPC tube has a label specifying the CCN for the sample being loaded in that tube. After loading the sample, transfer the label on the culture tube to the receiving flask to complete the sample loading. THEN continue to the next sample.

11.3.3 Collect each sample extract in a 250 mL Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation. Monitor sample volumes collected.

11.3.3.1 The volume of GPC eluate collected for each sample extract processed through GPC cleanup may be used to indicate problems with the system during sample processing. The GPC chemist will note any collected volume variations in the GPC Run/Maintenance Log (Attachment 4) as well as any corrective action taken.

11.3.3.2 Changes in sample volumes collected may indicate one or more of the following problems.

- Change in solvent flow rate caused by channeling in the column or changes in column pressure.

- Increase in column operating pressure due to the adsorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

- Leaks in the system or significant variances in room temperature.

11.4 Final Concentration and Exchange of Extract

11.4.1 The GPC fractions collected are transferred to a Kuderna-Danish (K-D) setup containing a 10 mL concentrator tube, 500 mL K-D evaporative flask, and a 3-ball macro-sampler column. Concentrate the extracts on a water bath set at 80 - 90° C. Adjust the vertical position of the apparatus and water temperature as required to concentrate the extract in 15 - 30 minutes. Remove the extracts from the water bath when their volume is approximately 4 mL. Add 60 mL of hexane to the K-D apparatus and mix thoroughly by tilting and shaking the K-D. Return the extracts to a 90 - 95° C water bath and concentrate to an apparent volume of 4 mL. Remove them from the water bath and allow them to cool. Remove the concentrator tube and further concentrate the hexane extract to 2.0 mL using nitrogen evaporation on the N-EVAP by Organomation. Alternatively, microsnyder concentration may be used.

CAUTION: Do not allow the hexane extract to concentrate below 1.0 mL at any time during the solvent concentration step. Doing so may cause low surrogate recoveries.

11.4.2 The 2.0 mL of extract is now ready for Sample Preparation Procedure-938, "Manual Florisil Cartridge Cleanup of Water and Soil Extracts for the Analysis of Pesticide/PCB by CLP.

11.4.3 Return the extracts to the GPC chemist for the required Florisil cleanup. Florisil clean-up is required for pesticide/PCB extracts under the OLM04.3 SOW and for pesticide extracts under the SOM01.1 SOW.

Note: Any samples that were loaded into two or more loops must be recombined before concentrating.

12.0 Data Analysis & Calculations

Calculations must be consistent with the Quality Contol SOP 13.6 "Numerical Data Reduction".

13.0 Method Performance

This method was validated in conjunction with analytical methods through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP SOW OLM04.3**, SOM01.1 (May, 2005)**

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3640A

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.5 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.6 Quality Control SOP 13.6 "Proper Documentation Procedures"

16.7 Quality Control SOP 13.4 "Numerical Data Reduction"

16.8 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.9 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.10 NELAC Standards, June 2003

16.11 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.12 New York State Environmental Laboratory Approval Program, Certification Manual, December 2005

16.13 CompuChem Quality Manual, Revision 7, Update 1, 12/13/2005

16.14 Sample Control SOP 4.1, "Receiving Samples"

16.15 Sample Control SOP 4.6, "Storing Samples"

16.16 Sample Preparation Procedure –722 (-1014 → -938): Preparation of Water Samples for the Analysis of Pesticides/ PCBs by CLP and NYSASP

16.17 Sample Preparation Procedure-726 (-213 → -940 → -938): Preparation of S/S/S Samples for the Analysis of Pesticides/PCBs (EPA CLP + NYSASP)

16.18 Sample Preparation Procedure –069, "Sample Preparation for Pesticides/PCBs in Water by SW-846 and NYSASP"

16.19 Sample Preparation Procedure –733 (-169 → -940) Low Level Preparation for Analysis of Pesticides/PCBs in Sediment/Soil (SW-846 + NYSASP)

16.20 Sample Preparation Procedure-938, "Manual Florisil Cartridge Cleanup of Water and Soil Extracts for the Analysis of Pesticide/PCB by CLP, SW-846, and NYSASP"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 - GPC Weekly Calibration Log

17.2 Attachment 2 – Pest/PCB Weekly GPC Calibration Check

17.3 Attachment 3 - Extraction Worksheet (-743)

17.4 Attachment 4 - GPC Run/Maintenance Log

Attachment 1

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GPC Weekly Calibration Log: ABC #4

Tape a copy of the U/V trace in the space below. Make copies of this page and attach them to the associated extraction sheets. Label each peak on the U/V trace with the compound name. Show resolution calculations.

Date/Time: _____ Room Temp: _____ Technician Name/ID: _____

GPC calibration standards (listed in order of elution)	**Time** (min) Or (CM)	**RSD** (%)
Corn oil		
Bis(2-ethylhexyl)phthalate	_____	_____
Methoxychlor		
Perylene	_____	_____
Sulfur		

Lot No. of GPC Calibration Standard Used _____

Acceptance criteria: ± 5.0% change from previous calibration (Do not round.)
Retention time criteria met. Y N

Calculate and record retention time shift from the previous calibration/UV trace.

Flow Rate: 5.0 ml/min Column: Bio-Beads Chart Speed: 15 cm/hr

Column Pressure: _____psi

Pest/PCB		**Semivolatiles**
Dump Time: _____min	> 85% resolution between corn oil and phthalate Y N	Dump Time: _____min
Collect Time: _____min	> 85% resolution between phthalate and methoxychlor Y N	Collect Time: _____min
Collect Volume: _____ml	> 90% resolution between perylene and sulfur Y N	Collect Volume: _____ml
Wash Time: 10:00 min		Wash Time: _____ min

Reviewed By: _____ **Date:** _____

4/7/00:mlj

Attachment 2

COMPUCHEM

A Division of Liberty Analytical

PEST/PCB WEEKLY GPC CALIBRATION CHECK

USEPA CLP SOW
-940

TECHNICIAN NAME: _____ DATE RAN: _____

EMPLOYEE ID: _____ GPC INSTRUMENT #: ABC GPC # _____

SAMPLE ID NUMBER*	GPC INJ. VOLUME	FINAL VOLUME	COMMENTS
A 1A	2.0 ml	10.0 ml	#4033 PESTICIDE SPIKE SOLUTION
A 1B			LOT #
A 2	2.0 ml	10.0 ml	1016/1260 PCB MIXTURE
			LOT #
A 3	2.0 ml	5.0 ml	METHYLENE CHLORIDE BLANK
			MANUFACTURER & LOT #

* COMPLETE SAMPLE ID NUMBER USING GPC COLUMN NUMBER ALONG WITH MONTH AND DATE, (i.e., A402022, A402022, & A40202B)

RUN WEEKLY GPC CALIBRATION THROUGH GPC CLEANUP EVERY SEVEN CALENDAR DAYS AND SUBMIT TO GC LAB FOR ANALYSIS.

A_____1A = 4.0 mL OF SPIKING SOLUTION DILUTED TO 10.0 mL MeC12 (for OLM04.3 SOW)

A_____1B = 800 µL OF SPIKING SOLUTION DILUTED TO 10.0 mL MeC12 (for SOM01.1 SOW)

A_____2 = A MIXTURE OF 2 µg OF AROCLOR 1016 AND 2 UG OF AROCLOR 1260 IN A FINAL VOLUME OF 10.0 mL WITH METHYLENE CHLORIDE (for the OLM04.3 SOW only)

A_____B = METHYLENE CHLORIDE ONLY

SUPERVISOR REVIEW: _____

FINAL VOLUME VERIFIED: _____

EXTRACTS RECEIVED BY: _____

5/05/06:jad

Attachment 3

COMPUCHEM a Division of Liberty Analytical

ASSIGNED TO: _____ **EXTRACTION WORKSHEET** **DATE EXTRACTED/POSTED:**

EPA CLP SOW SOM01.1

EMP ID NUMBER: _____ **EPA LOW LEVEL PEST S/S/S** **BATCH NO.:**

	COMPUCHEM NUMBER	CLIENT SAMPLE ID	QC SAMPLE TYPE	-743 SAMPLE WEIGHT (g)	FIANL EXTRACT VOL. (ml)	GPC FINAL VOL. (ml)	COMMENTS
1					10.0		
2					10.0		
3					10.0		
4					10.0		
5					10.0		
6					10.0		
7					10.0		
8					10.0		
9					10.0		
10					10.0		
11					10.0		
12					10.0		
13					10.0		
14					10.0		
15					10.0		
16					10.0		
17					10.0		
18					10.0		
19					10.0		
20					10.0		
21					10.0		
22					10.0		
23					10.0		
24					10.0		
25					10.0		
26					10.0		

				SURROGATE & SPIKE ADDED BY
	SURROGATE	No.	449	_____ / _____
		Amt.	1.0 mL	INITIALS DATE
		Lot #		
	MATRIX SPIKE	No.	4027	
		Amt.	1.0 mL	
		Lot #		
	LCS SPIKE	No.	4039	
		Amt.	1.0 mL	
		Lot #		

FINAL VOLUME VERIFIED _____

SUPERVISOR REVIEWED _____

Witness _____/_____
Initials Date

GPC Run Date:_____ Weekly GPC Calib. ID#:_____ Florisil Cart. Run Date:_____ Florisil Lot #:_____

Analysts Initials: Extracted_____ KD_____ N2_____ Bottle up_____

Manufacturer and lot number of reagents/solvents used _____

Attachment 4

ABC GPC #4 Run/Maintenance Log
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GPC Run Date: _____ **Analyst:** _____

Position	Sample ID	Position	Sample ID
1		13	
2		14	
3		15	
4		16	
5		17	
6		18	
7		19	
8		20	
9		21	
10		22	
11		23	
12			

Dump Time:_____

Collect Time: _____

Wash Time: _____

Head Pressure: _____

o GCP tubes cleaned prior to use
o Position cleaned prior to use
o Viewed first sample uptake
oFilled in GPC data on worksheet

Date of corn oil calibration _____

For Pests, date of weekly standards _____

For SV, date of GPC blanks _____

Comments/observations _____

_____

_____

_____

_____

Maintenance performed, if any

Reviewed By: _____

Date: _____

6/29/01:dce

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

ACCREDITED IN ACCORDANCE WITH

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 477 SOP Section #: 2.5.27 Revision #: 10

SOP Title: GCMS Analysis of Extractable Semivolatiles in Aqueous and Solid Sample Extracts by SW-846

Effective date: (QA fills in) 11/29/06

- Procedure prepared by: Rospass Date: 11/9/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Andrew McLean Pottinger Date: 11/29/06
- Reason for change: removed reference to endrin in DFTPP; added update LCS and surrogate control limits
- This procedure meets the requirements of the following approved method references: SW-846 3rd Edition, Update III, method 8270C, 12/96

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 477: GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Sample Extracts by SW-846

Table of Contents

Instrument Procedure 477: GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Sample Extracts by SW-846

1.0 Scope and Application

This method covers the determination of a variety of semivolatile compounds in aqueous and solid samples. These compounds are partitioned into an organic solvent and are amenable to analysis by gas chromatograph/mass spectrometer (GC/MS) instrumentation. The method involves solvent extraction of the matrix, and GC/MS analysis to determine the semivolatile compounds present in the sample. Target compounds for this method are listed in Attachment 1, along with associated internal standards, surrogates and quantitation ions. Reporting limit are included in Attachment 3.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Water samples are extracted prior to analysis following Sample Preparation Procedure –079, "Preparation of Water Samples for the Analysis of Low Level Semivolatile Organic Compounds by SW-846 and NYSASP." Soil samples are extracted using Sample Preparation Procedure –176, "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Low-Level Semivolatile Organic Compounds SW-846 and NYSASP," Sample Preparation Procedure –236, "Soxhlet Extraction of S/S/S Samples by Method 3540C in SW-846 & NYSASP," Sample Preparation Procedure –183, "Medium Level Preparation Procedure for Semivolatile Organics in Soil Samples by SW-846 and NYSASP," or Sample Preparation Procedure –247, "Automated Soxhlet Extraction of Soil/Sediment/Sludge and Wipe Samples by SW-846 + NYSASP."

Sample extracts are injected into a GC/MS system equipped with a narrow bore, fused silica capillary column. The GC is temperature programmed in order to separate the analytes for detection by the MS. Target compounds are identified in the samples by analyzing standards under the same conditions used for samples, comparing mass spectra with established library spectra, and comparing GC retention times with retention times from the latest continuing calibration verification standard.

Internal standards and surrogate compounds are added to all samples, QC and standards. An average relative response factor is established for each target and surrogate compound during the initial calibration procedures and this average response factor is used to calculate the analyte concentrations.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

To meet the requirements of the DoD-QSM, if the reporting limit is not at least three times higher than the calculated MDL value, the reporting limit is adjusted upward in order to achieve this minimal ratio.

3.3 Reporting Units – µg/L for water and µg/Kg for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days per client request) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, and matrix spike duplicate must also be prepared together at a rate of 5% for DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

3.6 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, the following sample must be evaluated for carryover.

4.2 If any contamination is present in blanks, samples or QC, the source must be identified and re-extraction of part or the entire analytical batch may be required.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, i.e., extraction, safety glasses, gloves and lab coats are a minimum requirement. Sample extracts should be prepared under a hood. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Analytical Columns

6.1.1 RTX-5MS, 0.32 mm ID, 0.25 μm film thickness

6.1.3 RTX-5MS, 0.25 mm ID, 0.25 mm film thickness

6.2 GC

6.2.1 Hewlett Packard (HP) 5890 Series with Electronic Pressure Control (EPC)

6.2.2 HP 6890 Series with EPC

6.3 MS

6.3.1 HP 5972A Mass Selective Detector (MSD)

6.3.2 HP 5973 MSD

6.4 AutoSampler

6.4.1 Hewlett Packard 7673 and 6890 Automated Liquid Sampler

6.5 Data System

6.5.1 A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. HP ChemServer is used for data collection.

6.5.2 The data processing computer has software that searches any GC/MS data file for ions of a specified mass and plots ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Also, for the non-target compounds, the software compares sample spectra against reference library spectra. The reference library used is the NIST Mass Spectral library (NBS129K.1)

6.5.3 The Hewlett Packard HP 9000 series 735 Unix Workstation employing with Target3 software by Thru-Put Systems is used for data processing and short term data storage.

6.6 Syringes

6.6.1 10 µL Hamilton syringe

7.0 Reagents & Standards

Refer to the Standards Preparation Logbook 22 F and applicable standard preparation SOPs for details on preparation of standards used in this procedure. All standards used in this procedure are prepared in optima grade methylene chloride.

7.1 Reagent water – All water used in this procedure must be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (19^{th} and 20^{th} Editions of Standard Methods, Method 1080), and is demonstrated to meet the blank criteria contained in this Standard Operating Procedure (SOP). It is referred throughout this SOP as **reagent** water.

7.2 Methylene Chloride – analytical grade

7.3 Reference Standards

7.3.1 Stock standards

7.3.1.1 Stock standards are prepared from certified, pure standard materials or purchased as certified solutions.

7.3.1.2 Stock standards are stored as recommended by the manufacturer (4 ± 2° C) in the Organic Standards Preparation laboratory.

7.3.1.3 Stock standards are replaced after 1 year or sooner, if problems occur.

7.3.2 Working Standards

Working standards are stored in the analysis laboratory at 4 ± 2° C separate from sample extracts, when not in use. Working standards are prepared every six months or sooner.

7.3.2.1 Internal standard solution is prepared at a concentration of 800 µg/mL.

7.3.2.2 Initial calibration standard solutions are prepared at concentrations of 5, 10, 25, 40, 60 and 80 µg/mL.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be analyzed within 40 days of extraction.

8.3 Prior to analysis, all extracts must be stored in the freezer at -10^{o} C in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogates are added to all samples, standards, and QC samples and are used to measure the efficiency and accuracy of the analytical system. Statistical control limits for surrogates are listed in the following table.

Compound	Aqueous (% Recovery)	Soil (% Recovery)
Nitrobenzene-d_5	**35-110**	**28-110**
2-fluorobiphenyl	**45-110**	**39-112**
Terphenyl-d_{14}	**49-120**	**51-119**
Phenol-d_5	10-110	**38-110**
2-fluorophenol	**11-110**	**33**-110
2,4,6-tribromophenol	**44-131**	**34-150**

9.1.1.1 If the surrogate recoveries are outside the control limits in 9.1.1, re-analyze the sample. If the surrogate recoveries are still outside of control limits, re-extract and reanalyze the sample.

9.1.1.2 If insufficient sample volume remains for re-extraction or the holding time has been exceeded, report results and document the failing surrogate results in the narrative.

9.1.2 The surrogate control limits required by the DoD-QSM are listed in the following table.

Compound	Aqueous (% Recovery)	Soil (% Recovery)
Phenol-d_5	N/A	40-100
2-Fluorobiphenyl	50-110	45-105
Terphenyl-d_{14}	50-135	30-125
2,4,6-Tribromophenol	40-125	35-125
2-Fluorophenol	20-110	35-105
Nitrobenzene-d_5	40-110	35-100

9.1.2.1 If the surrogate recovery control limits in 9.1.2 are not met and sufficient volume remains, re-extract and reanalyze the sample.

9.1.2.2 If field sample results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "J" flag.

9.1.2.3 If QC samples results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "Q" flag.

9.2 Internal Standards

9.2.1 Internal standard is added to an aliquot of the extract just prior to analysis in the instrumentation laboratory and is used in the quantitation of target analytes and surrogates.

9.2.2 The internal standard retention times in the continuing calibration standard must not differ more than ± 30 seconds from those in the mid point standard (80 ηg) in the most recent initial calibration. Additionally, the area response of the internal standards in the continuing calibration verification standard must be within -50% and +100% of those in the mid point standard of the most recent initial calibration.

9.2.3 If a sample fails internal standard acceptance criteria, reanalyze.

9.3 Method Blanks

9.3.1 A method blank is prepared with every analytical batch of up to 20 samples. For SC DHEC, the method blank is performed at a frequency of 10%. It is used to indicate extraction efficiency and contamination control within the analytical system. The method blank must be analyzed on each GC/MS system used to analyze the associated samples.

9.3.2 Any target analyte detected in the method blank must be less than the reporting limit. Except for SC DHEC, phthalates are allowed to be present at up to five times the reporting limit. Surrogate and internal standard responses must be within acceptance windows

9.3.2.1 To meet the requirements of the DoD-QSM, all target analytes in the method blank must be at concentrations < half the reporting limit.

9.3.3 If the method blank fails acceptance criteria for surrogate recovery or contamination, the analytical system is considered to be out of control. The source of the contamination must be investigated and appropriate corrective measures taken and documented before more samples are analyzed. All samples processed with a method blank that is out of control must be re-extracted and reanalyzed, unless the method blank meets acceptance criteria upon re-injection.

9.4 Laboratory Control Sample

9.4.1 An LCS for aqueous matrix is **reagent** water and for solid matrix is furnaced Ottawa sand, or an additional portion of furnaced sodium sulfate,

which has been fortified with target compounds and surrogate standards. The LCS is prepared with each extraction batch of up to 20 samples. For SC DHEC, the laboratory control sample is performed at a frequency of 10%. The LCS should be analyzed after any method blank associated with the group of samples. The LCS data are used to ensure that any failing spiked target compound in the matrix spike and matrix spike duplicate is due to interference and not representative of an analytical system that is out of control.

9.4.4 **An acceptable LCS will contain all project compounds within the control limits listed in Attachments 5. A certain number of recoveries may be outside of the control limits but within the marginal exceedances listed in Attachment 5 depending on the number of compounds spiked. See the following table.**

Number of analytes in the LCS	Allowed number of Marginal Exceedances
>90	**5**
71-90	**4**
51-70	**3**
31-50	**2**
11-30	**1**
<11	**0**

9.4.5 To meet the requirements of the DoD-QSM, the LCS must meet the control limits listed in Attachment **6** with allowance for a number of marginal exceedances based on the number of compounds spiked **(see table in section 9.4.4)**. All target compounds must be spiked.

9.4.5.1 If the LCS fails acceptance criteria, re-extract and reanalyze all associated samples (if sufficient sample volume remains).

9.4.2.2 Sample results associated with a failing LCS must be qualified in the narrative. Refer to the DoD-QSM "Q" flag.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared with each SDG. For SC DHEC, the matrix spike/matrix spike duplicate are performed at a frequency of 10% for water samples and 5% for soil samples.

9.5.2 The majority of the acid spiked compound recoveries and relative percent differences (RPDs) and the majority of the base/neutral spiked compound recoveries and RPDs should be within the control limits listed in Attachment **5 and the RPD limits listed in Attachment 7**. A subset of analytes may be reported depending on specific project requirements.

9.5.2.1 To meet the requirements of the DoD-QSM, the LCS control limits must be applied to the duplicate matrix spikes. The RPD between the duplicate matrix spikes must be ≤ 30%,

9.5.2.2 If the duplicate matrix spikes do not meet DoD-QSM criteria, contact the client for guidance. Results for specific compounds in the original sample associated with failing matrix spikes must be qualified in the narrative as estimated values. Refer to the DoD-QSM "J" flag.

9.5.3 If the sample and associated MS/MSD show the same recovery trend, then re-extraction is not required. If the original associated with the MS/MSD does not meet QC criteria, it should be reanalyzed, or re-extracted then reanalyzed, if the MS/MSD surrogate recoveries are within limits.

9.5.4 If LCS is acceptable, and the MS/MSD results are consistent with the original unspiked sample, it can be assumed that the poor recovery is matrix related.

9.6 Duplicates

9.6.1 Duplicates, at a frequency of 10% for water samples and 5% for soil samples, are required when processing samples submitted to meet the regulatory requirements of SC DHEC. This may be satisfied with the MS/MSD.

9.7 Contingency

9.7.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.7.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.7.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.7.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 Tuning the GC/MS

10.1.1 Prior to analysis of any samples, blanks, or calibration standards, the laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for a tuning standard performance check solution containing Decafluorotriphenylphosphine (DFTPP).

10.1.1.1 At the time of tuning the instrument, the laboratory clock and instrument system clock must be verified to be in agreement for both time and date. Document that the lab's clock and date (LCD) are the same as the instrument's system clock and date (SCD) by writing "LCD=SCD" in the instrument's run log.

10.1.2 The instrument is standardized (tuned) by analyzing the DFTPP tuning solution every 12 hours. Two µL of the 25 ηg/µL DFTPP solution (50 ηg) are injected into the GC/MS.

10.1.3 The peak selection criteria for DFTPP analysis are as follows.

10.1.3.1 Acquire and average the apex of the DFTPP peak and one scan immediately before and after the apex. Subtract a single background scan prior to the peak, but no more than 20 scans prior to the elution of DFTPP. Also, do not subtract part of the DFTPP peak. The DFTPP acceptance criteria are listed in the following table.

Mass	Ion Abundance Criteria
51	30.0-80.0% of mass 198
68	< 2.0% of mass 69
69	present
70	< 2.0% of mass 69
127	25.0-75.0% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance(see note)
199	5.0-9.0% of mass 198

Mass	Ion Abundance Criteria
275	10.0-30.0% of mass 198
365	Greater than 0.75% of mass 198
441	Present but < mass 443
442	40.0-110.0% of mass 198
443	15.0-24.0% of mass 442

Note: All ion abundances are normalized to m/z 198, the normal base peak.

10.1.4 The DFTPP solution also contains benzidine, pentachlorophenol, **and** 4,4'-DDT to assess the condition of the column and injection port.

10.1.4.1 Benzidine, pentachlorophenol, and 4,4´-DDT are added as evaluation aids to indicate injector port and GC column efficiencies.

10.1.4.2 4,4´-DDT **is** used to assess the instrument condition prior to GC/MS confirmations of pesticide and PCB analytes.

10.1.4.3 Excessive 4,4´-DDT breakdown and poor peak shape and response for benzidine and pentachlorophenol indicate maintenance is required for the injector port or GC column.

10.1.5 The following breakdown and tailing factor criteria must be met for the DFTPP analysis to be acceptable.

10.1.5.1 The % breakdown for DDT into DDE and DDD must not exceed 20%.

10.1.5.2 The tailing factor for benzidine must not exceed 3.0.

10.1.5.3 The tailing factor for pentachlorophenol must not exceed 5.0.

Note: When the breakdown or tailing factor criteria are not met it is indicative that instrument maintenance is necessary for the column and/or the injector port.

10.1.6 All valid injections must be made within the 12-hour calibration period that begins with the time of the injection of the DFTPP. Each injection thereafter must be recorded on the instrument run log (Attachment **3**).

10.2 Initial Calibration

10.2.1 An initial six-point calibration ("multipoint") must be performed if a valid multipoint for the method has not already been analyzed on the instrument or if the continuing calibration standard (12-hour continuing calibration standard) does not meet all acceptance criteria.

10.2.2 The initial calibration standards are at six concentration levels (10, 20, 50, 80, 120, and 160 total ηg per 2 µL). Each calibration standard contains each compound of interest and each surrogate.

10.2.3 Allow the calibration standard solutions to equilibrate to room temperature. Place an aliquot of each calibration standard solution in an amber 2 mL autosampler vial and place the vials on the instrument's autosampler.

Note: All injection volumes are 2 µL and are performed with a using cold needle.

10.2.4 Initial Calibration Acceptance Criteria

10.2.4.1 The System Performance Check Compounds (SPCCs) N-Nitroso-di-n-propylamine, 2,4-Dinitrophenol, Hexachlorocyclopentadiene, and 4-Nitrophenol must have an average response factor of ≥ 0.050.

10.2.4.2 The Calibration Check Compounds (CCCs) Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, Acenaphthene, Pentachlorophenol, Diphenylamine, 2,4,6-Trichlorophenol, Fluoranthene, Di-n-octylphthalate and Benzo(a)pyrene have a maximum RSD of 30%.

10.2.4.3 Each of the remaining compounds must have an average RSD of ≤ 15% for the initial calibration to be acceptable.

Note: For samples submitted to meet the regulatory requirements of the State of South Carolina, the non-CCC target analytes must not exceed 50% RSD.

10.2.5 Alternate Initial Calibration Acceptance Criteria

If the initial calibration criteria in 10.2.4 are not met, the calibration options in section 10.2.5.1 or 10.2.5.2 are used.

10.2.5.1 A linear or quadratic regression is used. The correlation coefficient must be ≥ 0.99 for the calibration curve to be acceptable. To meet the requirements of the DoD-QSM the linear regression correlation coefficient must be ≥ 0.995.

Note: Neither the SC DHEC nor the DoD-QSM accept the use of quadratic regression.

10.2.5.2 The "grand mean" is used. The mean of the RSD for all of the compounds present in the calibration standard is calculated and must be ≤ 15%. The client must be provided with a list of the individual compounds that did not meet the ≤ 15% RSD criteria.

Note: Calibration using the "grand mean" is not allowed by the SC DHEC or the DoD-QSM.

10.2.6 Corrective Actions for the Initial Calibration

10.2.6.1 Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the column, or take other corrective action to achieve the technical acceptance criteria.

10.2.6.2 Compare responses for the analytes in each of the standard levels to verify that a single standard analysis is not presenting results significantly higher or lower then the other standard analyses. If that is the case, the wrong standard may have been injected. Or the standard prepared incorrectly. Reanalyze the standard and calculate the response factors and % RSD.

10.2.6.3 The calibration range may be narrowed to determine if linearity can be achieved. The highest or lowest calibration standard may be replaced with a lower or higher concentration. This may cause more dilution re-analyses, if the high level standard is replaced or change the reporting limit if the lower standard is replaced.

10.2.6.4 If neither of the corrective actions in section 10.2.6.2 or 10.2.6.3 produce an acceptable calibration, perform instrument maintenance and analyze a new initial calibration.

10.3 Initial Calibration Verification

10.3.1 Inject 2 µL of second source ICV standard immediately after the initial calibration has been completed.

10.3.2 All analytes must be in the range of ± 30% of the expected value. An allowance is made that 10% of the analytes can be within 40% of the expected value.

10.3.2.1 To meet the requirements of the DoD-QSM, all analyte recoveries in the ICV must be within ± 25% of the expected value.

10.3.3 If criteria in **10.3.2** or **10.3.2.1** are not met, reanalyze the ICV standard. If the ICV criteria are still not met, investigate and correct the problem, and analyze a new initial calibration.

10.4 Continuing Calibration Verification

10.4.1 Inject 2 µL of continuing calibration verification (CCV) standard at the beginning of the 12-hour period after the DFTPP acceptance criteria have been met. The CCV standard is the mid-level initial calibration standard level (40 µg/mL).

10.4.2 CCV Acceptance Criteria

10.4.2.1 The internal standard retention times for the CCV standard must not differ from the corresponding internal standards in the mid-point standard (80 ηg) in the most recent initial calibration by more that ± 0.5 minutes (30 seconds).

10.4.2.2 The internal standard area responses for the CCV standard must be with ± 50 % of the corresponding internal standard area responses in the mid-point standard of the most recent initial calibration.

10.4.2.3 The SPCCs (see 10.2.4.1) must have a minimum response factor of ≥ 0.050.

10.4.2.4 The CCCs (see 10.2.4.2) must have a percent drift/difference of ≤ 20.

10.4.2.5 The remaining target compounds do not have defined % difference/drift criteria. The lab has established a warning limit of 50% and an action limit of 90% D. These criteria are based strictly on established U.S. EPA data validation guidelines where values greater than 90% result in rejection of data.

Note: For samples submitted to meet the regulatory requirements of the SC DHEC, the non-CCC have a warning limit of 40% D and an action limit of 50%D.

10.4.3 If continuing calibration acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration is performed.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The run log (Attachment **3**) must contain the date and time of the injection, the volume of the extract injected, standard identification numbers, sample identification numbers, case/SDG numbers, any comment relevant to the injection, and any preventive maintenance performed. Preventive maintenance includes front-end maintenance, rear end maintenance, and tuning the instrument. Any major maintenance, i.e., changing the source or column, is recorded in the instrument maintenance logbook.

11.1 Calibrate the GC/MS as outlined in section 10.0

11.2 Instrument settings

11.2.1 GC Conditions

- **Injector** **Split/Splitless**
- **Carrier Gas** **Helium**
- **Injection Port Temp (°C)** **280 – 320**
- **Sweep Flow (ml/min)** **100**
- **Column Flow (ml/min)** **1.5 – 2.7**
- **Initial Temp (°C)** **40**
- **Hold (min)** **2**
- **Ramp Rate (°C/min)** **15 – 25**
- **Final Temp (°C)** **305 – 315**
- **Hold (min)** **2 – 7**
- **Transfer Line Temp (°C)** **280 – 310**

Note: GC conditions may vary with regular instrument maintenance.

11.2.2 Mass Spectrometer Scanning parameters

- **Low Mass: 35 amu**
- **High Mass: 500 amu**
- **Threshold: 150 amu**
- **Scan time: ≤ 1 second**
- **Electron energy: 70 volts (nominal)**

11.**3** Sample Analysis

11.**3**.1 Allow the sample extracts to equilibrate to room temperature. Aliquot 50 µL of methylene chloride into an autosampler vial. Add 50 µL of the sample extract and 2.5 µL of internal standard solution (800 ηg/µL). Close each vial with a crimp-top cap containing a Teflon® septa. Transfer vials to the instrument's autosampler for analysis. All injections are made using the cold needle injection technique. The injection volume is 2.0 µL and this will yield 40 ηg per 2 µL on column of each of the internal standards.

11.**3**.2 Type the sample information into the autosampler sequence in HP ChemServer and start the analyses. All valid injections must be made within the 12-hour calibration period that begins at the moment of injection of the DFTPP. The DFTPP and each injection thereafter must be recorded on the instrument run log (Attachment **3**).

11.**4** Sample Processing

11.**4**.1 Process each sample by entering the sample preparation information from the extraction sheet into the corresponding field on the Target3 (Einstein) data system.

11.**4**.2 If any of the internal standards, surrogates, or spike compounds are missing or failing, check the peak integration in "Target Review". Changes can be made in Target Review and EICPs can be generated. Any compound for which manual peak integration has been performed will have an "M" flag displayed on the quantitation report. These entries must be assigned a numerical code and the analyst's initials and the date must appear on each quantitation report page containing an "M" flag.

11.**4**.3 The GC/MS analyst has the initial review responsibility. The **analyst** must ensure that the internal standard, surrogate recovery, and spike

recovery acceptance criteria are met for each field and QC sample and that all target analyte concentrations are within the calibration range.

11.**4**.4 If internal standards do not meet acceptance criteria in section 9.0, the sample must be re-injected to confirm a matrix effect or discount a miss-injection.

11.**4**.5 If surrogates do not meet acceptance criteria in section 9.0 and there are no problems with the surrogate or internal standard solutions, the sample must be scheduled for re-extraction and reanalysis. Re-extraction is not needed, if the sample was used and the original for duplicate matrix spikes and those analyses produced similar surrogate recoveries.

11.**4**.6 If the on-column amount of any target analyte exceeds the initial calibration range, the extract must be diluted, internal standard added, and the sample re-injected.

11.**5**. Target Compound Identification

11.**5**.1 The mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria.

- All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.
- The relative intensities of ions specified above must agree within ± 20% between the library and sample spectra.
- Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for.

11.**5**.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory will report that identification.

11.**6** Tentatively Identified Compound (TIC) Identification

11.**6**.1 TIC compounds are identified by comparing the mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library. TIC compounds are quantified by comparing the MS response of the peak from the reconstructed ion chromatogram (RIC) to the MS response for a peak

produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis & Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ \mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} x\ 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax\ x\ C(is)}{A(is)\ x\ Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured
A(is) = Area of the characteristic ion (EICP) for the specific internal standard
C(is) = Concentration of the internal standard (in µg/l)
Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.9 Concentration

12.9.1 Concentration of aqueous samples by GC/MS analysis

$$\mu g / L = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(\overline{RRF})(Vo)(Vi)}$$

where: Ax = area of the characteristic ion from the EICP for the compound to be measured
Ais = area of the characteristic ion for the EICP for the internal standard
Is = amount of internal standard injected (ηg)
$\overline{RRF}$ = mean relative response factor from initial calibration standards

Vo = volume of water extracted (mL)
Vi = volume of extract injected (µL)
Vt = volume of the concentrated extract (µL)
Df = dilution factor. If no dilution, Df = 1.0

12.9.2 Concentration of soil samples (dry weight basis) by GC/MS

$$\mu g / kg = \frac{(Ax)(Is)(Vt)(Df)(2.0)}{(Ais)(\overline{RRF})(Vi)(Ws)(D)}$$

where: Ax, Ais, Is, Vt, Vi, $\overline{RRF}$, and Df are the same as given for water
2.0 = GPC factor (if used)
Ws = weight of sample extracted, in grams
D (dry weight)= $\frac{100 - \% \text{ moisture}}{100}$

12.9.3 Concentration of water and soil samples (dry weight basis) by GC/MS using quadratic (second order) fit in Target:

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where:
b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
x = area of analyte/area of Internal Standard
n = amount of Internal Standard
y = amount in ng on column
Rsp = area of analyte/area of Internal Standard

Example:
Area of acenapthene = 35659
Area of IS = 613275
b = -0.0909161
m^1 = 9.605304
m^2 = 7.132688
ηg of IS = 250
response = 35659/613275 = 0.058145

Amount in ηg on column =

$$(250 ng)\left[-0.0909161 + 9.605304 x\ 0.058145 + 7.132688 x\ 0.058145\text{^}2\right] = 1229 ng$$

$$Concentration\ (water)\ \mu g / L = \frac{(y)(Df)(Vt)}{(Vo)(Vi)}$$

$$Concentration\ (soil)\mu g / Kg = \frac{(y)(Df)(Uf)(Vt)}{(Vi)(Ws)(D)}$$

where:

Uf = unit of correction for GPC, if used

12.9.4 Concentration of soil samples (dry weight basis) by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where: As = Area of the target analyte peak in the sample
Ais = Area of the internal standard peak
Cs = Concentration of the target analyte on column
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

$$Concentration\ (\text{water})\mu g / L = \frac{(Cs)(Df)(Vt)}{(Vo)(Vi)}$$

$$Concentration\ (\text{soil})\ \mu g / Kg = \frac{(Cs)(Df)(Uf)(Vt)}{(Vi)(Ws)(D)}$$

12.9.5 Concentration of TICs

$$TIC\ Amount\ (\text{water})\ \mu\text{g/L} = \frac{(Area\,TIC)\ x\ Amount(Std)(Df)(Vt)}{(Area\ IS)\ x\ 1(RF)(Vo)(Vi)}$$

$$TIC\ Amount\ (\text{soil})\ \mu\text{g/Kg} = \frac{(Area\,TIC)\ x\ Amount(Std)(Df)(Uf)(Vt)}{(Area\ IS)\ x\ 1(RF)(Vi)(Ws)(D)}$$

where: Area (TIC) = area response from RIC for non-target compound
Amount (Std) = amount of internal standard added to the sample, in µg/L
Area (IS) = area response of the nearest internal standard in the reconstructed ion chromatogram
1(RF) = assumed response factor of 1

12.10 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is generally performed using 100 µL sample plus 900 µL diluent for a total volume of 1.0 mL. It should be recorded on the run log as "10x."

12.11 Percent Breakdown of DDT in DFTPP Solution

$$\%\ DDT = \frac{DDD + DDE\ (Summation\ of\ degradation\ peak\ areas)}{DDT + DDD + DDE\ (Summation\ of\ all\ peak\ areas)}\ x\ 100$$

12.12 Tailing Factor for Benzidine and Pentachlorophenol

12.12.1 See the diagram, Attachment **4**.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. **See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.**

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, 3rd Edition, Update 3, December, 1996, Method 8270C

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.**7** NELAC Standards, June 2003

16.**8** QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.**9** CompuChem Quality Manual, Revision 7, Update 1, December 13, 2005, plus revisions

16.**10** Sample Control SOP 4.6, "Storing Samples"

16.**11** Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006

17.0 Attachments as Tables, Diagrams, Flowcharts

17.1 Attachment 1 – Target Compounds, Corresponding Internal Standards, and Quantitation Ions

17.2 Attachment 2 – Additional Appendix IX Analytes

17.3 Attachment 3 – Semivolatile Instrument Run Log

17.4 Attachment 4 - Peak Tailing Factor Demonstration

17.5 Attachment 5 - In-house LCS Control Limits

17.6 Attachment **6** – DoD-QSM LCS Control Limits

17.8 **Attachment 7 - Matrix Spike Relative Percent Differences**

Attachment 1

Target Compounds, Corresponding Internal Standards, and Quantitation Ions

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
1,4-dichlorobenzene-d_4 *	1	152	150, 115
2-Fluorophenol $	1	112	64, 92
Phenol-d_5 $	1	99	71, 42
N-Nitrosodimethylamine	1	42	74, 43
Pyridine	1	79	52, 78
Benzaldehyde	1	77	105,106
Nitrosomethylethylamine	1	88	56, 71
N-Nitrosodiethylamine	1	102	56, 57
Phenol	1	94	66, 65
Pentachloroethane	1	167	165, 117
bis(2-Chloroethyl)ether	1	93	95, 63
2-Chlorophenol	1	128	130, 92
1,3-Dichlorobenzene	1	146	148, 111
1,4-Dichlorobenzene	1	146	148, 111
Benzyl alcohol	1	108	107, 79
1,2-Dichlorobenzene	1	146	148, 111
2-Methylphenol	1	108	90, 107
2,2'-oxybis[1-chloropropane]	1	45	121, 123
3/4-Methylphenol	1	108	107, 79
Acetophenone	2	105	120, 77
N-Nitroso-di-n-propylamine	1	70	130, 113
Hexachloroethane	1	117	119, 166
Naphthalene-d_8 *	2	136	68, 137
Nitrobenzene-d_5 $	2	82	128, 54
Nitrobenzene	2	77	123, 93
Isophorone	2	82	138, 95
2,4-Dimethylphenol	2	107	91, 77

Attachment 1 (continued)

Target Compounds, Corresponding Internal Standards, and Quantitation Ions

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
2-Nitrophenol	2	139	92, 65
bis(2-Chloroethoxy)methane	2	93	95, 123
2,4-Dichlorophenol	2	162	164, 178
1,2,4-Trichlorobenzene	2	180	182, 145
Naphthalene	2	128	129, 92
4-Chloroanaline	2	127	129, 92
2,6-Dichlorophenol	2	162	164, 126
4-Chloro-3-methylphenol	2	107	144, 77
N-Nitroso-di-n-butylamine	2	84	99, 116
Hexachlorobutadiene	2	225	227, 260
2-Methylnaphthalene	2	141	142, 115
1-Methylnaphthalene	2	142	141, 115
Acenaphthene-d_{10} *	3	164	162, 181
2-Fluorobiphenyl $	3	172	171, 174
2,4,6-Tribromophenol $	3	330	332, 141
Hexachlorocyclopentadiene	3	237	239, 235
2,4,6-Trichlorophenol	3	196	200, 97
2,4,5-Trichlorophenol	3	196	200, 97
2-Chloronaphthalene	3	162	164, 127
2-Nitroaniline	3	65	138, 92
1,3-Dinitrobenzene	3	168	122, 75
Dimethyl phthalate	3	163	194, 164
1,2-Dinitrobenzene	3	168	63
2,6-Dinitrotoluene	3	165	148, 121
Acenaphthylene	3	152	151, 153
3-Nitroaniline	3	138	92, 65
Acenaphthene	3	154	153, 152
2,4-Dinitrophenol	3	184	107, 91
4-Nitrophenol	3	109	93, 65
2,4-Dinitrotoluene	3	165	89, 119

Attachment 1 (continued)

Target Compounds, Corresponding Internal Standards, and Quantitation Ions

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
Dibenzofuran	3	168	139, 169
Diethyl phthalate	3	149	177, 105
4-Chlorophenyl phenyl ether	3	204	206, 141
Fluorene	3	166	165, 139
4-Nitroaniline	3	138	108, 65
Phenanthrene-d_{10} *	4	188	94, 80
4,6-Dinitro-2-methylphenol	4	198	168, 121
N-Nitrosodiphenylamine[1]	4	169	168, 167
4-Bromophenyl phenyl ether	4	248	250, 141
Hexachlorobenzene	4	284	286, 142
Pentachlorophenol	4	266	268, 230
Phenanthrene	4	178	176, 89
Anthracene	4	178	176, 89
Methyl parathion	4	109	125, 263
Di-n-butyl phthalate	4	149	150, 278
Fluoranthene	4	202	101, 200
Chrysene-d_{12} *	5	240	120, 236
Benzidine	5	184	185, 156
Pyrene	5	202	101, 200
Butylbenzyl phthalate	5	149	206, 91
3,3'-Dichlorobenzidine	5	252	254, 154
Bis(2-ethylhexyl) phthalate	5	149	167, 150
Benzo(a)anthracene	5	228	226, 114
Chrysene	5	228	226, 114
Perylene-d_{12} *	6	264	260, 265
Terphenyl-d_{14} $	5	244	122, 212
Di-n-octyl phthalate	6	149	150, 279
Benzo(b)fluoranthene	6	252	126, 250
Benzo(k)fluoranthene	6	252	126, 250
Benzo(a)pyrene	6	252	126, 250
Indeno(1,2,3-c,d)pyrene	6	276	138, 274
Dibenzo(a,h)anthracene	6	278	139, 276
Benzo(g,h,i)perylene	6	276	138, 274

$ Surrogate

* Internal Standard

Attachment 2

Appendix IX Compounds (Additional non-routine/project-specific analytes)

Compounds	Internal Standard	Primary Quantitation Ions	Secondary Quantitation Ions
Ethylmethacrylate	1	69	99, 114
2-Picoline	1	93	66, 92
Nitrosomethylethylamine	1	88	42, 43
Methyl methanesulfonate	1		79, 65
n-Nitrosodiethylamine	1	102	56, 42
Ethyl methanesulfonate	1	79	109, 97
Aniline	1	93	66, 65
Pentachloroethane	1	167	165, 117
Benzyl alcohol	1	108	77, 79
n-Nitrosopyrrolidine	1	100	41, 68
n-Nitrosomorpholine	1	116	86, 56
o-Toluidine hydrochloride	1	100	107, 89
n-Nitrosopiperidine	1	114	55, 42
Benzoic acid	2	122	105, 77
O,o,o-Triethylphosphorothioate	2	198	170, 115
2,6-Dichlorophenol	2	162	164, 126
Hexachloroprene	2	213	215, 211
n-Nitroso-di-n-butylamine	2	84	57, 41
p-Phenylenediamine	3	108	80, 54
Safrole	2	162	104, 77
1-Methylnaphthalene	2	142	141, 115
1,2,4,5-Tetrachlorobenzene	3	216	218, 214
Isosafrole	3	162	104, 131
1,4-Naphthoquinone	3	158	102, 130
1,3-Dinitrobenzene	3	168	122, 75
Pentachlorobenzene	3	250	252, 215
1-Naphthylamine	3	143	115, 89
2,3,4,6-Tetrachlorophenol	3	232	234, 166
2-Naphthylamine	3	143	115, 116
Zinophos	3	97	143, 107
5-Nitro-o-toluidine	3	152	106, 77
Diphenylamine	4	169	168, 167
1,2-Diphenylhydrazine	3	77	182, 105
Sulfotepp	3	322	202

Attachment 2 (continued)

Appendix IX Compounds (Additional non-routine/project-specific analytes)

Compounds	Internal Standard	Primary Quantitation Ions	Secondary Quantitation Ions
1,3,5-Trinitrobenzene	4	75	213, 120
Diallate (trans isomer)	4	86	234, 43
Phorate	4	75	121, 97
Phenacetin	4	108	109, 179
Diallate (cis isomer)	4	86	234, 43
Dimethoate	4	87	125, 93
4-Aminobiphenyl	4	169	168, 170
Pentachloronitrobenzene	4	237	295, 142
Pronamide	4	173	175, 145
Disulfoton	4	88	97, 89
Methyl parathion	4	109	125, 263
Parathion	4	109	97, 291
4-Nitroquinoline-1-oxide	4	190	128, 75
Methapyrilene	4	97	191, 71
Isodrin	4	193	195, 263
p-Dimethylaminoazobenzene	5	225	120, 77
Chlorobenzilate	5	251	139, 253
Famfur	5	218	93, 125
3,3'-Dimethylbenzidine	5	212	106, 180
2-Acetylaminofluorene	5	181	180, 223
7,12-Dimethylbenz(a)anthracene	6	256	239, 241
3-Methylcholanthrene	6	268	252, 126
1,3,5-Trinitrobenzene	4	75	213, 120
Diallate (trans isomer)	4	86	234, 43
Phorate	4	75	121, 97
Phenacetin	4	108	109, 179
Diallate (cis isomer)	4	86	234, 43
Dimethoate	4	87	125, 93
4-Aminobiphenyl	4	169	168, 170
Pentachloronitrobenzene	4	237	295, 142
Pronamide	4	173	175, 145
Disulfoton	4	88	97, 89
Methyl parathion	4	109	125, 263
Parathion	4	109	97, 291

Attachment 3

COMPUCHEM a division of Liberty Analytical Corp DATE ____/____/____ INITIAL TIME OF TUNE_____ SHIFT/S(A)_____(B)_____(C)____
GC/MS SEMIVOLATILE RUN LOG TIME TUNE EXPIRES_____ LINKER /METHOD_____
COMPUCHEM LOGBOOK 4 YYY 13 (5972hp64)
PREVENTIVE MAINTENANCE_____

	INJECTED	FILE NAME	REPORTED	DATE	TIME	CLIENT ID#	CASE/SDG#	AMOUNT INJECTED	CHEMIST	COMMENTS(ETC.)/DISPOSITION
1				/ /						
2				/ /						
3				/ /						
4				/ /						
5				/ /						
6				/ /						
7				/ /						
8				/ /						
9				/ /						
10				/ /						
11				/ /						
12				/ /						
13				/ /						
14				/ /						
15				/ /						
16				/ /						
17				/ /						
18				/ /						
19				/ /						
20				/ /						
21				/ /						
22				/ /						
23				/ /						
24				/ /						

STANDARDS

	Tune	Analytical	Int. Std.	Column Type
Std. ID #	_____	_____	_____	_____
Lot #	_____	_____	_____	

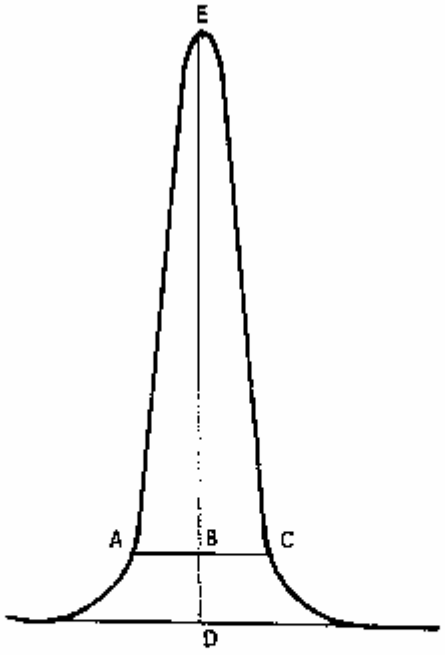
SUPERVISOR APPROVAL_____
DATE _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's has SOP occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Solvent Lot # _____

12/28/05:jad

Attachment 4



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11} = 1.1$

Tailing factor calculation.

Attachment 5

In-House LCS Recovery Limits

Semivolatile Compound	Soil (% Recovery)	Marginal Exceedance (% Recovery)
N-Nitrosodiphenylamine	**20-118**	10-**135**
Pyridine	**20**-100	10-**160**
Benzaldehyde	**20**-100	10-**160**
Phenol	**38-112**	**26-123**
bis (2-Chloroethyl)ether	**30-112**	**16-125**
2-Chlorophenol	**30-100**	**10-160**
1,3-Dichlorobenzene	**21-104**	**10-118**
1,4-Dichlorobenzene	**21-104**	**10-118**
Benzyl alcohol	**40-107**	**29-118**
1,2-Dichlorobenzene	**24-108**	**10-122**
2-Methylphenol	**42-110**	**30-121**
2,2'-Oxybis (1-chloropropane)	**24-107**	**10-121**
Acetophenone	**32-105**	**20-117**
3/4-Methylphenol	**38-114**	**26-127**
n-Nitroso-di-n-propylamine	**23-130**	**10-147**
Hexachloroethane	**20-105**	**10-120**
Nitrobenzene	**20-135**	**10-156**
Isophorone	**39-114**	**27-126**
2-Nitrophenol	**33-119**	**19-133**
2,4-Dimethylphenol	**43-117**	**31-130**
bis (2-Chloroethoxy)methane	**39-107**	**24-118**
2,4-Dichlorophenol	**42-119**	**29-132**
1,2,4-Trichlorobenzene	**32-112**	**19-126**
Naphthalene	**38-118**	**24-132**
4-Chloroaniline	26-**129**	10-**146**
Hexachlorobutadiene	**31-118**	**16-132**
Caprolactam	**45-122**	**32-134**
4-Chloro-3-methylphenol	**46-120**	**34-132**
2-Methylnaphthalene	**39-111**	**27-123**
1-Methylnaphthalene	**39-112**	**27-124**
Hexachlorocyclopentadiene	**20-134**	**10-160**
2,4,6-Trichlorophenol	**46-127**	**33-141**
2,4,5-Trichlorophenol	**52-122**	**40-134**
1,1'-Biphenyl	42-**116**	**30-126**
2-Chloronaphthalene	**11-112**	**33-123**
2-Nitroaniline	**50-109**	**40-118**

Attachment 5 (continued)

In-House LCS Recovery Limits

Semivolatile Compound	Soil (% Recovery)	Marginal Exceedance (% Recovery)
Dimethylphthalate	**50-120**	**39-132**
2,6-Dinitrotoluene	47-**119**	**35-131**
Acenaphthylene	**50**-125	**38-137**
3-Nitroaniline	**43-100**	**34-160**
Acenaphthene	**50-126**	**37-138**
2,4-Dinitrophenol	**20-121**	10-**140**
4-Nitrophenol	**26-145**	**10-160**
2,4-Dinitrotoluene	**50-125**	**37-137**
Dibenzofuran	**47-116**	**36-127**
Diethylphthalate	**51-123**	**39-135**
4-Chlorophenyl-phenylether	**52-123**	**40-135**
Fluorene	**52-128**	**40-141**
4-Nitroaniline	**42-100**	**32-160**
4,6-Dinitro-2-methylphenol	**32-127**	**16-143**
n-Nitrosodiphenylamine	**46-125**	**33-125**
1,2-Diphenylhydrazine	**48-110**	**38-110**
4-Bromophenyl-phenylether	**53-122**	**42-134**
Hexachlorobenzene	**49-126**	**36-139**
Atrazine	**22-100**	**10-160**
Pentachlorophenol	**35-132**	**19-148**
Phenanthrene	**51-129**	**38-143**
Carbazole	**45-135**	**30-150**
Anthracene	**53-136**	40-**149**
di-n-Butylphthalate	**49-121**	**37-133**
Fluoranthene	**56-134**	**43-147**
Benzidine	**20-150**	10-**160**
Pyrene	**48-136**	**34-151**
Butylbenzylphthalate	**42-137**	**36-153**

Attachment 5 (continued)

In-House LCS Recovery Limits

Semivolatile Compound	Soil (% Recovery)	Marginal Exceedance (% Recovery)
3,3'-Dichlorobenzidine	**21-100**	**10-160**
Bis (2-Ethylhexyl) phthalate	**37-133**	**21-149**
Benzo(a)anthracene	**57-134**	**44-147**
Chrysene	**53-135**	**39-148**
Di-n-Octylphthalate	**43-138**	**26-154**
Benzo(b)fluoranthene	**48-130**	**35-143**
Benzo(k)fluoranthene	**61-141**	**47-155**
Benzo(a)pyrene	**60-131**	**48-142**
Indeno(1,2,3-cd)pyrene	**47-137**	**32-152**
Dibenz(a,h)anthracene	**51-127**	**38-139**
Benzo(g,h,i)perylene	**43-129**	**29-142**

Note: LCS recovery limits are based on in-house performance statistics. A subset of analytes may be reported, depending on project requirements. While the limits were statistically derived, from actual laboratory data, it is expected that a minimum of 40% recovery is achieved for the vast majority of analytes.

Attachment 5 (continued)

Semivolatile Compound	Aqueous (% Recovery)	Marginal Exceedance (% Recovery)
N-Nitrosodiphenylamine	20-100	10-120
Pyridine	20-100	10-120
Benzaldehyde	20-100	10-117
Phenol	20-100	10-120
bis (2-Chloroethyl)ether	31-100	20-120
2-Chlorophenol	23-100	15-120
1,3-Dichlorobenzene	24-100	13-120
1,4-Dichlorobenzene	25-100	15-120
Benzyl alcohol	29-100	19-120
1,2-Dichlorobenzene	27-100	16-101
2-Methylphenol	25-100	13-120
2,2'-Oxybis (1-chloropropane)	24-100	13-120
Acetophenone	39-100	29-105
3/4-Methylphenol	24-100	15-120
N-Nitroso-di-n-propylamine	34-101	23-112
Hexachloroethane	28-100	18-120
Nitrobenzene	20-131	10-150
Isophorone	43-105	32-115
2-Nitrophenol	39-103	28-113
2,4-Dimethylphenol	19-109	10-120
bis (2-Chloroethoxy)methane	35-110	25-106
2,4-Dichlorophenol	39-103	28-113
1,2,4-Trichlorobenzene	36-101	25-111
Naphthalene	40-104	29-114
4-Chloroaniline	25-115	10-130
Hexachlorobutadiene	29-103	17-115
Caprolactam	20-100	10-120
4-Chloro-3-methylphenol	41-103	30-113
2-Methylnaphthalene	40-100	30-110
1-Methylnaphthalene	39-100	29-109
Hexachlorocyclopentadiene	20-123	10-143
2,4,6-Trichlorophenol	46-113	35-124
2,4,5-Trichlorophenol	41-119	29-132
1,1'-Biphenyl	49-109	36-119
2-Chloronaphthalene	46-105	36-115
2-Nitroaniline	34-107	22-119

Attachment 5 (continued)

Semivolatile Compound	Aqueous (% Recovery)	Marginal Exceedance (% Recovery)
Dimethylphthalate	**53-116**	**43-127**
2,6-Dinitrotoluene	**51-115**	**40-126**
Acenaphthylene	**50-115**	**39-126**
3-Nitroaniline	**20-110**	**10-125**
Acenaphthene	**53-118**	**43-128**
2,4-Dinitrophenol	**20-102**	**10-118**
4-Nitrophenol	**10-100**	**10-120**
2,4-Dinitrotoluene	**52-119**	**41-131**
Dibenzofuran	**50-107**	**41-129**
Diethylphthalate	**52-118**	**45-111**
4-Chlorophenyl-phenylether	**51-117**	**40-128**
Fluorene	**53-121**	**41-132**
4-Nitroaniline	**20-110**	**10-129**
4,6-Dinitro-2-methylphenol	**38-116**	**25-129**
N-Nitrosodiphenylamine	**52-117**	**41-127**
1,2-Diphenylhydrazine	**38-105**	**27-117**
4-Bromophenyl-phenylether	**51-115**	**41-125**
Hexachlorobenzene	**52-116**	**41-126**
Atrazine	**20-100**	**10-120**
Pentachlorophenol	**20-100**	**10-150**
Phenanthrene	**56-121**	**45-131**
Carbazole	**39-115**	**27-127**
Anthracene	**55-127**	**43-139**
Di-n-butylphthalate	**51-114**	**41-124**
Fluoranthene	**58-124**	**47-134**
Benzidine	**20-150**	**10-160**
Pyrene	**48-131**	**34-145**
Butylbenzylphthalate	**41-118**	**28-131**

Attachment 5 (Continued)

Semivolatile Compound	Aqueous (% Recovery)	Marginal Exceedance (% Recovery)
3,3'-Dichlorobenzidine	**20-100**	**10-120**
Bis (2-Ethylhexyl) phthalate	**47-121**	**35-133**
Benzo(a)anthracene	**53-128**	**41-140**
Chrysene	**50-130**	**36-143**
Di-n-octylphthalate	**30-148**	**10-160**
Benzo(b)fluoranthene	**41-130**	**26-145**
Benzo(k)fluoranthene	**39-150**	**19-160**
Benzo(a)pyrene	**46-135**	**31-150**
Indeno(1,2,3-cd)pyrene	**47-138**	**32-154**
Dibenz(a,h)anthracene	**47-134**	**32-148**
Benzo(g,h,i)perylene	**42-132**	**27-147**

Attachment 6

LCS Control Limits for 8270C required by the DoD-QSM

Compound	Aqueous Recovery Limits	Aqueous Marginal Exceedance	Solid Recovery Limits	Solid Marginal Exceedance
n-Nitrosodiphenylamine	50-110	35-120	50-115	40-125
Phenol	0-115	0-135	40-100	30-110
Bis(2-chloroethyl]ether	35-110	25-120	40-105	25-115
2-Chlorophenol	35-105	25-115	45-105	35-115
1,3-Dichlorobenzene	30-100	20-110	40-100	30-110
1,4-Dichlorobenzene	30-110	20-110	35-105	25-115
Benzyl alcohol	30-110	15-125	20-125	10-140
1,2-Dichlorobenzene	35-100	20-115	45-95	35-105
2-Methylphenol	40-110	25-120	40-105	30-115
2,2'-oxybis[1-chloropropane]	25-130	10-150	20-115	10-130
3/4-Methylphenol	30-110	20-125	40-105	30-120
n-Nitroso-di-n-propylamine	35-130	20-145	40-115	30-125
Hexachloroethane	30-95	15-105	35-110	20-120
Nitrobenzene	45-110	35-120	40-115	30-125
Isophorone	50-110	40-125	45-110	30-125
2-Nitrophenol	40-115	25-125	40-110	30-120
2,4-Dimethylphenol	30-110	15-125	30-105	20-115
Bis[2-chloroethoxy]methane	45-105	35-115	45-110	30-120
2,4-Dichlorophenol	50-105	40-115	45-110	35-120
1,2,4-Trichlorobenzene	35-105	25-120	45-110	30-110
Naphthalene	40-110	30-115	40-105	30-120
4-Chloroaniline	15-110	10-125	10-95	0-110
Hexachlorobutadiene	25-105	15-115	40-115	25-130
4-Chloro-3-methylphenol	45-110	35-120	45-115	35-125
2-Methylnaphthalene	45-105	35-115	45-105	30-115
2,4,6-Trichlorophenol	50-115	40-125	50-115	40-120
2,4,5-Trichlorophenol	50-110	40-120	50-110	40-120
2-Chloronaphthalene	50-105	40-115	50-105	40-115
2-Nitroaniline	50-115	35-125	50-115	35-125
Dimethylphthalate	25-125	10-145	25-125	10-145
2,6-Dinitrotoluene	50-115	35-130	50-115	35-130
Acenaphthylene	50-105	40-115	50-105	40-115
3-Nitroaniline	20-125	10-145	20-125	10-145
Acenaphthene	45-110	35-120	45-110	35-120
2,4-Dinitrophenol	15-140	10-160	15-130	10-150
4-Nitrophenol	0-125	0-145	15-140	10-160
2,4-Dinitrotoluene	20-120	40-130	50-115	35-130
Dibenzofuran	55-105	45-115	50-105	40-110
Diethyl phthalate	40-120	30-130	50-105	40-125
4-Chlorophenyl-phenylether	50-110	40-120	45-115	35-125
Fluorene	50-110	40-120	50-110	40-115
4-Nitroaniline	35-120	20-130	35-115	20-125
4,6-Dinitro-2-methylphenol	40-130	25-145	20-135	10-155

Attachment 6 (continued)

Compound	Aqueous Recovery Limits	Aqueous Marginal Exceedance	Solid Recovery Limits	Solid Marginal Exceedance
1,2-Diphenylhydrazine	55-115	45-120	N/A	N/A
4-Bromophenyl-phenylether	50-115	40-125	45-115	35-130
Hexachlorobenzene	50-110	40-120	45-120	35-130
Pentachlorophenol	40-115	25-130	25-120	10-135
Phenanthrene	50-115	40-130	50-110	40-120
Carbazole	50-115	35-130	45-115	30-130
Anthracene	55-110	45-120	55-105	45-115
Di-n-butyl phthalate	55-115	45-125	55-110	45-120
Fluoranthene	55-115	45-125	55-115	45-120
Pyrene	50-130	35-140	45-125	35-135
Butyl benzyl phthalate	45-115	35-130	50-125	35-135
3,3'-Dichlorobenzidine	20-110	10-125	10-130	0-145
Bis[2-ethylhexyl]phthalate	40-125	30-140	45-125	35-140
Benzo[a]anthracene	45-120	35-130	50-110	40-120
Chrysene	55-110	45-120	55-110	45-120
Di-n-octyl phthalate	35-135	20-155	40-130	25-145
Benzo[b]fluoranthene	45-120	35-130	45-115	35-125
Benzo[k]fluoranthene	45-125	30-135	45-125	30-135
Benzo[a]pyrene	55-110	45-120	50-110	40-120
Indeno[1,2,3-cd]pyrene	45-125	30-140	40-125	25-140
Dibenz[a,h]anthracene	40-125	30-140	40-125	25-140
Benzo [g,h,i]perylene	40-125	25-135	40-125	25-140

Surrogate recovery limits for 8270C required by the DoD-QSM

Compound	Aqueous (% Recovery)	Soil (% Recovery)
Nitrobenzene-d_5	40-110	35-100
2-fluorobiphenyl	50-110	45-105
Terphenyl-d_{14}	50-135	30-125
Phenol-d_5	N/A	40-100
2-fluorophenol	20-110	35-105
2,4,6-tribromophenol	40-125	35-125

Attachment 7

Matrix Spike Relative Percent Differences

Semivolatile Compound	Aqueous	Solid
N-Nitrosodiphenylamine	50	50
Pyridine	50	50
Benzaldehyde	50	42
Phenol	46	39
bis (2-Chloroethyl)ether	46	39
2-Chlorophenol	42	37
1,3-Dichlorobenzene	50	47
1,4-Dichlorobenzene	50	46
Benzyl alcohol	41	36
1,2-Dichlorobenzene	50	42
2-Methylphenol	50	32
2,2'-Oxybis (1-chloropropane)	48	40
Acetophenone	47	41
3/4-Methylphenol	50	40
N-Nitroso-di-n-propylamine	50	42
Hexachloroethane	50	43
Nitrobenzene	44	37
Isophorone	50	40
2-Nitrophenol	47	39
2,4-Dimethylphenol	50	31
2,4-Dinitrophenol	50	46
4-Nitrophenol	50	35
2,4-Dinitrotoluene	44	34
Dibenzofuran	45	33
Diethylphthalate	45	31
4-Chlorophenyl-phenylether	47	33
Fluorene	49	32
4-Nitroaniline	50	42
4,6-Dinitro-2-methylphenol	50	38
N-Nitrosodiphenylamine	50	31
1,2-Diphenylhyhydrazine	50	28
4-Bromophenyl-phenylether	47	37
Hexachlorobenzene	42	32
Atrazine	50	31
Pentachlorophenol	50	48
Phenanthrene	50	33

Attachment 7 (continued)

Matrix Spike Relative Percent Differences

Semivolatile Compound	Aqueous	Solid
Anthracene	**50**	**31**
Carbazole	**50**	**32**
Di-n-butylphthalate	**40**	**29**
Fluoranthene	**50**	**36**
Benzidine	**50**	**50**
Pyrene	**50**	**31**
Butyl benzylphthalate	**48**	**27**
3,3'-Dichlorobenzidine	**50**	**50**
Bis (2-ethylhexyl)phthalate	**49**	**44**
Benzo(a)anthracene	**50**	**35**
Chrysene	**50**	**35**
Di-n-octylphthalate	**50**	**33**
Benzo(b)fluoranthene	**50**	**39**
Benzo(k)fluoranthene	**50**	**40**
Benzo(a)pyrene	**50**	**29**
Indeno(1,2,3-cd)pyrene	**50**	**29**
Dibenzo(a,h)anthracene	**50**	**31**
Benzo(g,h,i)perylene	**50**	**31**

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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-183 SOP Section #: 2.5.2.4 Revision #: 7

SOP Title: Medium Level Preparation Procedure for Semivolatile Organics in Soil Samples by SW846 + NYSASP

Effective date: (QA fills in) 2/20/03

- Procedure prepared by: T. Cl Date: 2/9/03
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Jane C. Ellmore Date: 2/20/03
- Reason for change: Yearly update
- This procedure meets the requirements of the following approved method references: NYSASP, 6/2000+rev.; NELAC Standards 6/2000 + rev; SW846, 3rd Ed, Update III, Method 3550B + 8270C

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date: _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 1/5/04

Annual Review—Signature: _____ Date: 2/10/05

Annual Review—Signature: _____ Date: 7/5/06

Sample Preparation Procedure -183: Medium Level Preparation Procedure for Semivolatile Organics in Soil Samples by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure -183: Medium Level Preparation Procedure for Semivolatile Organics in Soil Samples by SW-846 and NYSASP

1.0 Scope and Application

This procedure describes the preparation of medium level soil samples for semivolatile (SV) GC/MS analysis.

The method detection limits (MDL) and reporting limits are based on the low level soil MDLs and lowest calibration standard concentration, respectively. See Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A 2.0 gram sample aliquot is extracted by sonication methylene chloride then filtered, and concentrated.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements.

The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in reagents, solvents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free of interferences under the conditions of the analysis by preparing and analyzing laboratory reagent blanks.

4.2 Matrix interferences may be caused by contaminants that were inadvertently co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be done to determine the possible matrix interferences.

5.0 Safety

5.1 The degree of toxicity or carcinogenicity of the chemicals used in this method has not been determined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for ensuring staff awareness of Occupational Safety and Health Administration (OSHA) regulations regarding safe handling of chemicals used in this method. Additional material on laboratory safety is available for the analyst.

5.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens:

- benzo(a)anthracene
- benzidine
- 3,3′-dichlorobenzidine
- benzo(a)pyrene
- dibenz(a,h)anthracene
- N-nitrosodimethylamine

6.0 Equipment & Supplies

6.1 Ultrasonic Disrupter, having a minimum power wattage of 300 watts, with pulsing capability. Use a 1/8" tapered microtip attached to a 1/2" horn.

6.2 Sonabox, to be used with disruptor for decreasing cartation sound.

6.3 Disposable, graduated, transfer pipets- 0.5 mL, 1.0 mL.

6.4 Centrifuge tubes- 250 mL.

6.5 Powder Funnel, 10 cm. diameter with Whatman No. 41 filter paper, or equivalent.

6.6 Concentration tube- 10 mL, graduated.

6.7 Boiling chips- solvent extracted (silicon carbide or equivalent).

6.8 Water bath- heated and capable of temperature control to ± 5°C

6.9 Balance- platform, capable of weighing to nearest 0.01 g.

6.10 Nitrogen blow-down apparatus-Organomation or equivalent.

6.11 Vials- 2 mL, with screw-top, Teflon-lined septa.

6.12 Spatula- stainless steel or Teflon

6.13 Stirring rod - glass

7.0 Reagents & Standards

All standards are prepared by the Organic Standards chemist. Details for the preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.) Standards are stored separately from samples at 2-4.4 °C in the reach in unit in the laboratory when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 **Methylene chloride**- Fisher, pesticide grade

7.3 Sodium sulfate--Use only sodium sulfate labeled "FURNACED SODIUM SULFATE".

7.3.1 Furnace the sodium sulfate in a shallow tray prior to use for at least four hour in a 400^{o} C oven.

7.4 Surrogate and Spike Standards

7.4.1 Semivolatile surrogate solution #393

7.4.2 8270 validation spike

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be extracted within 14 days of collection.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2-4.4^{o} C in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with each extraction batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS, or blank spike, BS, or matrix blank spike for NYSASP) must be prepared with each extraction batch of up to 20 samples.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared for every sample delivery group (SDG).

9.4 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of North Carolina. The MS/MSD satisfy the duplicate requirement for the NC DENR.

10.0 Calibration & Standardization

10.1 Ensure the balance has been calibrated for the day prior to its use following the Quality Control SOP 13.16, "Top Loading Balance Calibration & Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The sample preparation technician must complete the extraction worksheet (Attachment 2). Any unused portions must be z'ed out. The laboratory supervisor reviews the completed worksheet for accuracy and completeness and then signs it. The worksheet accompanies the sample to the analytical laboratory. Include on the worksheet the manufacturer and lot number of the reagents/solvents used.

11.1 Preparation of Equipment

11.1.**1** Rinse each **piece of glassware** with methylene chloride. Empty the methylene chloride into a waste container and repeat the process two more times. If the glassware selected for use is wet, it must be rinsed with **methanol** before the methylene chloride rinses.

11.1.**2** Label each piece of glassware with the sample number it is to be used for and the preparation procedure (-183). Use orange labels.

11.2 Sample Preparation and Extraction

11.2.1 Decant and discard any water layer on the sediment sample. Mix thoroughly to achieve homogeneity. Discard any foreign objects such as sticks, stones, and leaves. Sample should be at room temperature.

11.2.2 Place a VOA vial on the platform balance and press the tare button. This will automatically subtract the weight of the vial. Using a spatula weigh 2.0 g of sample (to the nearest 0.1 g) into a VOA vial. Record the weight on the extraction worksheet.

11.2.3 Two additional aliquots of a sample must be weighed for the MS/MSD. If the client has not designated a sample to be used for QC, the laboratory selects one.

11.2.4 For the blank and LCS, weigh 2.0 g of sodium sulfate.

11.2.5 Add 2.0 g of sodium sulfate to each sample and QC and mix thoroughly.

11.2.6 Add 1.0 ml of surrogate #393 to all samples and QC.

11.2.7 Add 1.0 ml of the 8270 validation spike standard to the LCS and MS/MSD. Record the standard ID numbers, the lot numbers, and volume added on the extraction worksheet.

11.2.8 Add 8.0 ml of methylene chloride to the sample spikes and LCS, and 9.0 ml of methylene chloride to all other samples.

11.2.9 Sonify the samples for two minutes using a 1/8th-inch microtip. The output control setting should be at 5, mode switch at pulse, and percent duty cycle at 50%.

11.2.10 Filter the extracts through a glass wool plug and collect 5 ml of extract in a 15-ml centrifuge tube. It is important to collect exactly 5 ml of extract for proper surrogate recovery.

11.2.10.1 Alternatively, 5.0 ml of extract may be syringe filtered through a .45 μm membrane.

11.3 Extract Concentration

11.3.**1** Concentrate the **5 ml** extract to 0.5 ml volume using the nitrogen blowdown technique **or microsnyder concentration**. Transfer the entire 0.5 ml into a 2-ml amber autosampler vial labeled with the sample number, prep code (-183), and completion date. Note the final volume on the extraction worksheet.

11.3.**2** Complete the extraction worksheet, mark the meniscus level on the vials, and place the completed worksheet along with the samples on the shelf for supervisor review and approval.

11.3.**3** The extract is now ready for GC/MS analysis following Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW846 and NYSASP".

11.3.4.5 Deliver the extractions worksheet and extracts to the designated location in the semivolatile laboratory and complete the chain-of-custody documentation.

11.4 Work Area Cleanup

11.4.1 Empty all solid material into the appropriate waste container. Rinse all used glassware with methylene chloride and take the rinsed glassware to the glassware prep area for cleaning.

11.4.2 Do not pour any methylene chloride in the sinks.

11.4.3 Roll up all absorbent counter covers and place them in the trash can. Return any remaining raw samples to the sample cart. Transfer samples to the Sample Custodian under chain-of-custody.

11.4.4 Pour the waste solvent into the appropriate container.

11.4.5 Lay down new absorbent counter cover on the prep table.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 1) and precision and accuracy for single analyst (Attachment 3). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Methods 3550B and 8270C

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions.

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, June 2000, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **3**, 1**2**/**19**/0**2**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Quality Control SOP 13.16, "Top Loading Balance Calibration & Maintenance."

16.16 Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW846 and NYSASP"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Method Detection Limits

17.2 Attachment 2 - Extraction Worksheet (-183)

17.3 Attachment 3 – Single Analyst Capability Study

Attachment 1

Preparation Date: January 13, 2004			GCMS Method 3550B/8270C Soil Sonication Semivolatiles											
Analysis Date: January 14, 19, 2004						Using higher of three instrument MDLs								
Instrument: 5972hp60/64/66														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg
n-Nitrosodimethylamine	73.54	36.91	56.68	104.29	51.56	80.77	62.00	43.41	46.55	61.75	83.5	21.29	61.6	330
Pyridine	27.23	6.21	8.23	44.03	56.81	43.74	41.33	31.38	5.03	29.33	83.5	19.05	55.2	330
Benzaldehyde	37.45	61.98	82.18	51.16	69.25	58.32	55.08	86.02	26.74	58.69	166.5	19.24	55.7	330
Phenol	73.94	82.27	86.96	93.16	89.68	97.75	87.21	95.34	75.28	86.84	83.5	8.37	24.2	330
Bis(2-chloroethyl)ether	62.87	66.02	68.85	72.12	66.75	78.39	74.22	78.62	70.11	70.88	83.5	5.46	15.8	330
2-Chlorophenol	66.23	66.81	71.10	72.79	72.59	75.85	70.97	75.65	62.49	70.50	83.5	4.49	13.0	330
1,3-Dichlorobenzene	64.20	70.42	75.62	69.66	69.10	70.79	75.07	78.85	71.16	71.65	83.5	4.30	12.4	330
1,4-Dichlorobenzene	64.96	74.34	77.35	74.46	69.06	79.66	78.66	75.91	71.31	73.97	83.5	4.77	13.8	330
Benzyl alcohol	19.50	43.85	39.18	57.23	53.65	40.52	29.80	39.05	37.48	40.03	83.5	11.35	32.9	330
1,2-Dichlorobenzene	63.38	70.67	75.96	75.33	65.44	73.09	81.60	73.90	67.05	71.82	83.5	5.78	16.7	330
2-Methylphenol	54.43	67.63	74.78	83.93	82.13	76.71	67.76	74.58	58.62	71.17	83.5	10.00	29.0	330
2,2'-oxybis(1-Chloropropane)	56.97	59.97	63.06	61.97	68.85	72.86	64.51	71.07	60.49	64.42	83.5	5.40	15.7	330
Acetophenone	59.67	62.81	64.83	72.62	67.03	69.87	68.16	74.31	60.61	66.66	83.5	5.13	14.9	330
3-/4-Methylphenol	125.68	142.20	144.21	164.59	157.78	160.13	144.12	154.13	130.97	147.09	166.5	13.23	38.3	330
n-Nitroso-di-n-propylamine	60.94	62.89	77.11	68.54	62.65	80.12	81.32	71.36	57.86	69.20	83.5	8.76	25.4	330
Hexachloroethane	61.60	66.17	73.72	72.23	59.12	69.36	78.04	73.00	66.46	68.86	83.5	6.10	17.7	330
Nitrobenzene	88.31	105.32	102.21	113.21	103.54	103.45	95.20	115.00	91.72	102.00	83.5	9.01	26.1	330
Isophorone	71.44	75.80	82.66	91.87	84.34	88.08	80.08	93.51	73.74	82.39	83.5	7.85	22.7	330
2-Nitrophenol	51.75	61.16	72.06	68.23	69.17	71.70	63.38	75.27	55.94	65.41	83.5	7.92	22.9	330
2,4-Dimethylphenol	31.17	74.61	60.06	101.90	89.44	55.57	36.99	58.74	46.68	61.68	83.5	23.37	67.7	330
Bis(2-chloroethoxy)methane	61.62	71.47	71.46	80.74	75.31	75.30	73.16	80.41	66.62	72.90	83.5	6.13	17.7	330
2,4-Dichlorophenol	59.44	69.63	78.45	83.79	75.64	74.74	70.31	77.78	67.13	72.99	83.5	7.21	20.9	330
1,2,4-Trichlorobenzene	70.28	82.66	83.47	83.99	76.30	83.04	86.32	81.01	71.22	79.81	83.5	5.81	16.8	330
Naphthalene	62.57	75.46	77.48	77.25	70.85	75.46	77.32	72.12	66.44	72.77	83.5	5.31	15.4	330
4-Chloroaniline	51.13	107.88	106.36	131.95	124.51	111.02	60.50	106.91	99.04	99.92	166.5	27.02	78.3	330
Hexachlorobutadiene	75.40	74.95	79.96	85.87	79.30	90.72	76.91	90.30	75.81	81.02	83.5	6.33	18.3	330
Caprolactam	44.91	15.70	63.02	44.40	47.84	49.60	54.25	45.63	55.05	46.71	83.5	13.10	37.9	330
4-Chloro-3-methylphenol	78.00	78.72	94.01	99.18	97.95	83.18	83.05	95.61	78.24	87.55	83.5	8.98	26.0	330
2-Methylnaphthalene	71.62	73.41	78.65	83.14	81.57	83.86	80.57	87.77	72.51	79.23	83.5	5.64	16.3	330
1-Methylnaphthalene	77.50	79.53	88.83	92.05	91.04	91.16	85.07	93.01	75.77	86.00	83.5	6.76	19.6	330
Hexachlorocyclopentadiene	544.93	648.34	668.88	688.14	637.85	668.90	669.18	650.54	568.90	638.41	835	48.85	141.5	330
2,4,6-Trichlorophenol	56.28	56.66	61.12	71.59	72.14	66.28	65.79	68.88	52.43	63.46	83.5	7.15	20.7	330
2,4,5-Trichlorophenol	53.67	62.04	63.08	75.09	74.17	73.25	60.47	67.34	60.85	65.55	83.5	7.38	21.4	330
1,1'-Biphenyl	72.81	78.16	82.10	85.25	89.38	91.15	81.15	90.03	74.72	82.75	83.5	6.72	19.5	330

Attachment 1 (continued)

Preparation Date: January 13, 2004			GCMS Method 3550B/8270C Soil Sonication Semivolatiles											
Analysis Date: January 14, 19, 2004						Using higher of three instrument MDLs								
Instrument: 5972hp60/64/66														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg
2-Chloronaphthalene	74.71	75.64	86.21	90.74	90.46	88.67	82.97	88.79	77.04	83.91	83.5	6.54	18.9	330
2-Nitroaniline	58.02	65.00	71.12	72.14	76.42	72.84	68.38	78.00	63.37	69.48	83.5	6.44	18.6	670
Dimethylphthalate	54.98	64.82	67.72	78.14	68.80	66.99	70.31	65.25	58.38	66.15	83.5	6.70	19.4	330
2,6-Dinitrotoluene	59.61	64.33	76.00	83.89	81.42	73.18	74.99	73.70	66.03	72.57	83.5	7.94	23.0	330
Acenaphthylene	65.84	72.10	74.16	82.42	78.78	78.32	71.35	78.77	66.03	74.20	83.5	5.87	17.0	330
3-Nitroaniline	60.77	102.52	100.18	120.66	114.77	109.50	80.45	105.53	98.50	99.21	166.5	18.34	53.1	670
Acenaphthene	71.02	77.05	82.79	85.55	86.15	86.62	79.29	86.11	72.56	80.79	83.5	6.09	17.6	330
2,4-Dinitrophenol	212.71	327.15	360.93	384.16	380.38	380.85	366.65	361.80	325.15	344.42	835	53.95	156.2	670
4-Nitrophenol	178.78	210.41	239.96	282.02	260.89	225.23	222.65	247.16	215.20	231.37	334	30.29	87.7	670
2,4-Dinitrotoluene	51.19	58.75	59.21	75.40	75.44	69.99	63.14	67.46	57.37	64.22	83.5	8.42	24.4	330
Dibenzofuran	69.48	74.69	75.31	86.64	84.68	84.28	77.27	84.24	72.29	78.76	83.5	6.29	18.2	330
Diethylphthalate	72.32	80.45	83.98	95.71	96.08	89.06	83.29	88.78	75.36	85.00	83.5	8.27	23.9	330
4-Chlorophenyl-phenylether	61.65	68.02	75.24	82.91	75.06	75.61	76.42	73.51	63.32	72.42	83.5	6.81	19.7	330
Fluorene	57.92	67.69	71.79	80.99	74.69	72.61	74.54	68.57	64.00	70.31	83.5	6.73	19.5	330
4-Nitroaniline	89.86	106.49	122.11	153.03	146.46	145.06	113.59	126.30	114.29	124.13	166.5	20.84	60.4	670
4,6-Dinitro-2-methylphenol	56.37	59.15	65.64	95.18	79.79	72.98	65.12	80.29	57.75	70.25	166.5	12.94	37.5	670
n-Nitrosodiphenylamine	69.32	77.08	81.14	90.77	93.64	80.07	77.42	81.37	69.56	80.04	83.5	8.24	23.9	330
1,2-Diphenylhydrazine	72.81	77.24	84.73	94.84	98.72	88.31	88.59	93.47	79.56	86.47	83.5	8.66	25.1	330
4-Bromophenyl phenylether	55.10	66.68	70.72	77.22	72.96	73.75	67.68	65.41	60.21	67.75	83.5	6.94	20.1	330
Hexachlorobenzene	72.99	86.16	91.52	101.17	88.93	90.50	92.39	84.54	70.88	86.56	83.5	9.54	27.6	330
Atrazine	98.57	99.39	108.97	128.50	133.74	115.29	114.86	124.14	103.32	114.09	83.5	12.73	36.9	330
Pentachlorophenol	96.06	30.42	246.05	104.64	29.64	85.69	69.53	166.00	56.64	98.30	166.5	69.37	200.9	670
Phenanthrene	70.74	76.67	78.82	90.12	91.29	85.86	81.23	84.13	73.31	81.35	83.5	7.15	20.7	330
Anthracene	68.11	75.98	80.28	93.27	92.43	82.30	76.26	83.05	71.99	80.41	83.5	8.51	24.6	330
Carbazole	132.41	143.63	149.48	165.17	168.87	163.06	153.24	157.84	142.56	152.92	83.5	12.03	34.8	330
Di-n-butylphthalate	71.10	78.99	79.60	87.68	95.89	86.74	82.34	84.07	72.76	82.13	83.5	7.67	22.2	330
Fluoranthene	61.50	77.99	76.59	87.08	81.46	77.17	78.18	73.57	65.26	75.42	83.5	7.84	22.7	330
Pyrene	93.60	93.74	106.35	114.61	115.20	103.69	99.93	103.47	90.19	102.31	83.5	8.94	25.9	330
Butylbenzylphthalate	79.48	79.01	88.72	98.38	102.10	91.73	88.51	87.45	80.00	88.38	83.5	8.20	23.7	330
3,3'-Dichlorobenzidine	39.23	53.97	53.19	66.94	62.45	56.55	45.02	52.90	47.90	53.13	83.5	8.49	24.6	670
bis(2-ethylhexyl)phthalate	94.66	90.23	96.69	100.05	115.74	103.37	99.39	102.32	88.84	99.03	83.5	8.03	23.3	330
Benzo(a)anthracene	66.88	83.59	82.54	91.66	88.81	84.86	86.07	80.79	71.01	81.80	83.5	8.04	23.3	330
Chrysene	84.92	94.28	98.99	106.15	112.18	101.54	90.42	98.56	87.66	97.19	83.5	8.84	25.6	330
Di-n-octylphthalate	66.73	67.90	71.98	75.80	79.63	79.07	68.65	72.07	62.66	71.61	83.5	5.75	16.6	330
Benzo(b)fluoranthene	67.60	69.92	82.53	85.85	80.98	80.41	77.59	82.57	68.96	77.38	83.5	6.80	19.7	330
Benzo(k)fluoranthene	74.44	83.99	79.29	101.04	102.70	83.25	85.97	87.20	74.17	85.78	83.5	10.22	29.6	330
Benzo(a)pyrene	68.96	74.72	75.32	87.80	88.48	74.91	71.02	76.17	68.11	76.17	83.5	7.37	21.3	330
Indeno(1,2,3-c,d)pyrene	63.83	66.04	68.62	80.57	78.27	69.95	69.81	74.03	59.63	70.08	83.5	6.70	19.4	330
Dibenzo(a,h)anthracene	64.32	71.46	71.79	81.88	81.42	74.01	68.47	75.17	63.74	72.47	83.5	6.50	18.8	330
Benzo(g,h,i)perylene	67.90	68.50	75.59	79.48	81.67	72.91	68.19	73.19	61.97	72.16	83.5	6.21	18.0	330

Attachment 2

EXTRACTION WORKSHEET
Medium Level Soil S-V, Method 3550B for 8270C
CompuChem

ASSIGNED TO: _____ **-183**

EMP ID NUMBER: _____ **DATE EXTRACTED/POSTED:**_____

	SAMPLE NUMBER	QC SAMPLE TYPE	SAMPLE WEIGH (g)	FINAL VOLUME (ml)	COMMENTS
1					Comment: Only 50% of the extract is carried through the final volume
2					*Add 1.0 mL validation spike to LCS
3					
4					GPC (3640A) PERFORMED Y / N
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23		BLK			
24		LCS			

		S-VOL	Acid	B/N	Others
SURROGATE	NO.	393			
	AMT	1.0 ml			
	LOT				
SPIKE	NO.		3012	2021	VALID.
	AMT.		1.0 ml	1.0ml	1.0 ml
	LOT				

FINAL VOLUME VERIFIED:_____

SUPERVISOR REVIEWED:_____

Witness _____ / _____
Initials Date

SURROGATE & SPIKE ADDED BY: _____/_____
Initials Date

Analyst initials. Extracted _____ KD _____ N2 _____ Bottle up _____

Manufacturer and lot number of reagents/solvents used _____

Attachment 3

Laboratory Name/North Carolina Certificate Number: CompuChem/79															
Analyst: Gracure Bailey															
Study Date: November 29, 2001															
Method: 3550B/8270C Soil															
Compounds	TrueVal	Rep#1	Rep#2	Rep#3	Rep#4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	-3SD	+3SD	SOP	RSD
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	%R	%R	%R	of (x)	of (x)	% R	%R	%RSD	%
Bis(2-chloroethyl)ether	2667	2249	1914	2012	2000	2044	77	19-113	143.63	1612.9	2474.6	79	121	50	7
2,2'-oxybis(1-Chloropropane)	2667	2393	1780	1689	1600	1866	70	18-114	90.00	1595.5	2135.5	86	114	50	5
N-Nitroso-di-N-propylamine	2667	2275	2001	1895	2800	2243	84	12-140	404.53	1029.2	3456.3	46	154	50	18
Hexachloroethane	2667	2104	2044	1534	1800	1871	70	10-131	260.02	1090.4	2650.6	58	142	50	14
Nitrobenzene	2667	2415	2175	2358	2200	2287	86	18-127	117.67	1934.0	2640.0	85	115	50	5
Isophorone	2667	2391	2182	1861	2000	2109	79	21-117	229.66	1419.5	2797.5	67	133	50	11
Bis(2-chloroethoxy)methane	2667	2340	1989	1938	1900	2042	77	22-141	202.15	1435.3	2648.2	70	130	50	10
Naphthalene	2667	2112	2181	1946	2400	2160	81	14-113	188.10	1595.5	2724.0	74	126	50	9
4-Chloroaniline	2667	1926	1680	1949	1800	1839	69	10-107	124.46	1465.4	2212.1	80	120	50	7
Hexachlorobutadiene	2667	2095	2359	1814	2300	2142	80	11-137	246.20	1403.4	2880.6	66	134	30	11
2-Methylnaphthalene	2667	2225	2266	1948	2100	2135	80	16-146	143.13	1705.4	2564.1	80	120	50	7
Hexachlorocyclopentadiene	2667	2739	2856	1960	1700	2314	87	10-150	570.58	602.0	4025.5	26	174	50	25
2-Chloronaphthalene	2667	2371	2438	2124	2300	2308	87	15-138	135.14	1902.8	2713.7	82	118	50	6
2-Nitroaniline	2667	2321	2163	1874	2300	2165	81	20-132	205.95	1546.7	2782.3	71	129	50	10
Dimethylphthalate	2667	2353	2419	1946	2500	2305	86	20-129	246.44	1565.2	3043.8	68	132	50	11
2,6-Dinitrotoluene	2667	2514	2475	2079	2500	2392	90	18-131	209.29	1764.1	3019.9	74	126	50	9
Acenaphthylene	2667	2245	2380	2018	2500	2286	86	18-121	206.67	1665.7	2905.8	73	127	50	9
3-Nitroaniline	2667	2194	1870	1745	2000	1952	73	11-105	191.87	1376.6	2527.9	71	129	50	10
Acenaphthene	2667	2110	1584	2046	2400	2035	76	18-112	337.82	1021.5	3048.5	50	150	30	17
2,4-Dinitrotoluene	2667	2399	2494	1636	2400	2232	84	18-128	399.99	1032.3	3432.2	46	154	50	18
Dibenzofuran	2667	2254	2409	2210	2300	2293	86	24-131	85.47	2036.8	2549.7	89	111	50	4
Diethylphthalate	2667	2272	2584	2251	2400	2377	89	18-128	153.06	1917.6	2835.9	81	119	50	6
4-Chlorophenyl-phenylether	2667	2159	2348	1554	2500	2140	80	18-130	414.98	895.3	3385.2	42	158	50	19
Fluorene	2667	2206	2288	2210	2500	2301	86	15-123	137.93	1887.2	2714.8	82	118	50	6
4-Nitroaniline	2667	2113	2109	2033	2500	2189	82	10-113	210.74	1556.5	2821.0	71	129	50	10
N-Nitrosodiphenylamine	2667	2419	2272	2667	2300	2415	91	23-123	179.99	1874.5	2954.5	78	122	30	7
4-Bromophenyl-phenylether	2667	2338	2540	2395	2600	2468	93	21-131	122.25	2101.5	2835.0	85	115	50	5
Hexachlorobenzene	2667	2538	2634	2102	2600	2469	93	15-148	247.54	1725.9	3211.1	70	130	50	10
Phenanthrene	2667	2279	2292	2144	2500	2304	86	18-123	146.95	1862.9	2744.6	81	119	50	6
Anthracene	2667	2308	2262	2462	2700	2433	91	19-120	197.48	1840.6	3025.4	76	124	50	8
Carbazole	2667	2386	2407	2723	3500	2754	103	24-140	520.68	1192.0	4316.0	43	157	50	19
Di-n-butylphthalate	2667	2439	2959	2511	2300	2552	96	20-131	284.96	1697.4	3407.1	67	133	50	11
Fluoranthene	2667	2336	2417	2155	3000	2477	93	16-127	365.47	1380.6	3573.4	56	144	30	15
Pyrene	2667	2584	2078	2228	2800	2423	91	12-123	329.19	1434.9	3410.1	59	141	50	14
Butylbenzylphthalate	2667	2668	2172	2103	2300	2311	87	20-119	251.76	1555.5	3066.0	67	133	50	11
3,3'-Dichlorobenzidine	2667	1587	1287	1470	1900	1561	59	10-129	257.52	788.4	2333.6	51	149	50	16
bis(2-ethylhexyl)Phthalate	2667	2712	3257	2132	2200	2575	97	18-125	523.06	1006.1	4144.4	39	161	50	20
Benzo(a)anthracene	2667	2336	2195	2026	2900	2364	89	17-117	378.98	1227.3	3501.2	52	148	50	16
Chrysene	2667	2316	2144	2135	2900	2374	89	19-121	360.58	1292.0	3455.5	54	146	50	15
Di-n-octylphthalate	2667	2457	2061	1725	2000	2061	77	13-135	301.90	1155.0	2966.5	56	144	30	15
Benzo(b)fluoranthene	2667	2137	2042	1892	2700	2193	82	10-126	352.89	1134.1	3251.4	52	148	50	16
Benzo(k)fluoranthene	2667	2081	2070	1518	2500	2042	77	12-129	402.76	834.0	3250.5	41	159	50	20
Benzo(a)pyrene	2667	1971	1985	1714	2700	2093	78	15-120	423.73	821.3	3363.7	39	161	30	20
Indeno(1,2,3-c,d)pyrene	2667	1664	1771	1539	3500	2119	79	15-127	925.87	-659.1	4896.1	-31	231	50	44
Dibenzo(a,h)anthracene	2667	1747	2002	1747	3000	2124	80	10-130	596.24	335.3	3912.7	16	184	50	28
Benzo(g,h,i)perylene	2667	1842	1857	1692	3200	2148	81	13-125	705.44	31.4	4264.1	1	199	50	33

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-176 SOP Section #: 2.5.2.3 Revision #: 10

SOP Title: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Low-Level Semivolatiles by SW-846 and NYSASP

Effective date: (QA fills in) 3/19/04

- Procedure prepared by: Linda Carter Date: 1/15/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane C. Elkins Date: 3/1/04
- Reason for change: OH VAP compliance, Annual Review
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, Method 3550B and Method 8270C; NY State Analytical Services Protocol (NYSASP), June 2000, plus revisions

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date: _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] Date: 2/10/05

Annual Review—Signature: [signature] Date: 7/6/06

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -176: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Low-Level Semivolatiles by SW-846 and NYSASP

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Sample Preparation Procedure -176: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Low-Level Semivolatiles by SW-846 and NYSASP

1.0 Scope and Application

This procedure is used to determine the concentration of semivolatile organic compounds in solid samples following the procedure in SW-846 and NYSASP.

Method detection limits (MDL) and reporting limits are shown in Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A 30-gram aliquot of sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using sonication. The extract is separated from the sample by **buchner funnel filteration**. The sample is concentrated for analysis by GC/MS. Optional extract cleanup procedures may be employed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this

minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements.

The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/Kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers **(US ACE) and SC DHEC** do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **and 10% for SC DHEC**. If samples are batched together from different sites, project-specific QC must be processed.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in reagents, solvents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free of interferences under the conditions of the analysis by preparing and analyzing laboratory reagent blanks.

4.2 Matrix interferences may be caused by contaminants that were inadvertently coextracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be done to determine the possible matrix interferences.

5.0 Safety

5.1 The degree of toxicity or carcinogenicity of the chemicals used in this method has not been determined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for ensuring staff awareness of Occupational Safety and Health Administration (OSHA) regulations regarding safe handling of chemicals used in this method. Additional material on laboratory safety is available for the analyst.

5.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens:

- benzo(a)anthracene
- benzidine
- 3,3′-dichlorobenzidine
- benzo(a)pyrene
- dibenz(a,h)anthracene
- N-nitrosodimethylamine

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Ultrasonic Disrupter, having a minimum power wattage of 300 watts, with pulsing capability. Use a 3/4 "horn."

6.2 Sonabox, to be used with disruptor for decreasing cartation sound.

6.3 Disposable, graduated, transfer pipets- 0.5 mL, 1.0 mL.

6.4 Centrifuge tubes- 250 mL.

6.5 Powder Funnel, 10 cm. diameter with Whatman No. 41 filter paper, or Buchner funnel, or equivalent.

6.6 Kuderna-Danish (K-D) apparatus.

6.6.1 Concentration tube- 10 mL, graduated.

6.6.2 Evaporation flask- 500 mL, attached to concentrator tube with springs or clamps.

6.6.3 Snyder column- three ball macro.

6.7 Boiling chips- solvent extracted (silicon carbide or equivalent).

6.8 Water bath- Heated, capable of temperature control (± 5°C)

6.9 Balance- Platform, capable of weighing to nearest 0.01 g.

6.10 Nitrogen blow-down apparatus-Organomation or equivalent.

6.11 Vials- 2 mL, with screw-top, Teflon-lined septa.

6.12 Spatula- Stainless steel or Teflon

7.0 Reagents & Standards

Refer to the Standards Preparation Logbook 22 F for details on preparation of standards used in this procedure. All standards used in this procedure are prepared in optima grade methylene chloride. Standards are stored separately from samples in the laboratory when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type I water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methylene chloride - Fisher, pesticide-grade, methylene chloride is used for this analysis.

7.3 Acetone - pesticide-grade

7.4 Use methylene chloride and acetone to make 50/50 solution.

7.5 Sodium sulfate - Use only sodium sulfate labeled "FURNACED SODIUM SULFATE." Furnace the sodium sulfate in a shallow tray prior to use for at least four hour in a 400° C oven.

7.6 Surrogate and Spikes

7.6.1 Semivolatile surrogate solution #393.

7.6.2 8270 validation spike

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be extracted within 14 days of collection.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2-4.4° C in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with each extraction batch of up to 20 samples. **For SC DHEC, the method blank is performed at a frequency of 10%.**

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS, or blank spike, BS, or matrix blank spike for NYSASP) must be prepared with each extraction batch of up to 20 samples. **For SC DHEC, the LCS is performed at a frequency of 10%.**

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared for every sample delivery group (SDG). **For SC DHEC, the MS/MSD are also performed at a frequency of 5% for soil samples.**

9.4 Duplicates

9.4.1 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of **South** Carolina **DHEC**. The MS/MSD satisfy the duplicate requirement.

9.5 Contingency

9.5.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.5.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 Ensure the balance has been calibrated for the day prior to its use following the Quality Control SOP 13.16, "Top Loading Balance Calibration & Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The sample preparation technician must complete the extraction worksheet (Attachment 2). Any unused portions must be z'ed out. The laboratory supervisor reviews the completed worksheet for accuracy and completeness and then signs it. The worksheet accompanies the sample to the analytical laboratory. Include on the worksheet the manufacturer and lot number of the reagents/solvents used.

11.1 Preparation of Equipment

11.1.1 Rinse each of the items listed above with methylene chloride. Empty the methylene chloride into a waste container and repeat the process two

more times. If the glassware selected for use is wet, it must be rinsed with methanol before the methylene chloride rinses.

11.1.2 Label each piece of glassware with the sample number it is to be used for and the preparation procedure (in this case -176). Use orange labels.

11.2 Sample Preparation and Extraction

11.2.1 Decant and discard any water layer on a sediment sample. Mix thoroughly to achieve homogeneity, Discard any foreign objects such as sticks, stones, and leaves. Samples should be at room temperature. Place a 400 mL beaker, on the platform balance and press the tare button. This will automatically subtract the weight of the **beaker**. Using a spatula, weigh 30.0 g (± 0.5 g) of the sample in the beaker and record the exact weight of the sample to one decimal place on the extraction worksheet (Attachment 2).

11.2.2 Two additional 30 g portions from a designated sample are transferred to separate **beakers** for the matrix spike and matrix spike duplicate.

11.2.3 For the blank and LCS, weigh 90.0 g of sodium sulfate.

11.2.4 Add 60 g of furnaced sodium sulfate to each sample. Mix thoroughly to form a free-flowing powder.

11.2.5 Add 0.5 ml of surrogate standard #393 to each sample and QC with a 0.5 mL or 1.0 mL graduated pipet. Record the surrogate standard ID number, the lot number, and volume added on the extraction worksheet.

11.2.6 Using 1-ml pipets, add 1.0 ml each of the 8270 validation spike standard the MS/MSD and LCS. Record the standard ID numbers, the lot numbers, and volume added on the extraction worksheet.

11.2.7 Add 100 ml 1:1 methylene chloride/acetone to each sample and sonify for three minutes using a 3/4-inch horn. (100% power output with 50% duty cycle and 1-second pulse) Place the bottom of the horn about 1/2 an inch below the surface of the solvent, but above the sediment layer. Rinse the probe end in the **beaker** with a small amount of the 1:1 mixture. As each sample finishes sonification, **decant into a buchner funnel containing Whatman 41 filter paper. Use vacuum filtration and collect the extract in a sidearm flask.**

11.2.11 Repeat steps 11.2.7 two more times.

11.2.12 Upon completion of the third extraction, pour the entire sample into the **buchner** funnel and rinse with extraction solvent.

If particulate matter is observed, filter the entire extracted sample again through newly prepped filter paper using the original funnel or filter at a concentrated volume with a .45 µm membrane filter.

11.3 Extract Concentration

11.3.1 For each sample being concentrated, rinse a Snyder column with methylene chloride.

11.3.2 **Pour the extract into a KD apparatus.** Add 2-3 boiling chips to each K-D flask and attach a Snyder column. If the Snyder column is not still wet from the rinsing process, add 1-2 ml methylene chloride to the top of the column.

11.3.3 Place the K-D apparatus on a water bath set at 80-90°C. The apparatus should be placed in the bath so that the entire lower rounded surface of the flask is bathed in hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the sample reaches 2-4 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

11.3.4 Final concentration volume of semivolatile fractions:

11.3.4.1 Remove the Synder column and K-D, then concentrate the extract to 1.0 ml volume using the nitrogen blowdown technique or microsnyder concentraiton. Transfer the entire 1.0 ml into a 2-ml amber autosampler vial labeled with the sample number, prep code (-176), and completion date. Note the final volume on the Extractions Worksheet.

11.3.4.2 Deliver the Extractions Worksheet and extracts to the semivolatile laboratory and complete chain-of-custody documentation.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 1) and precision and accuracy for single analyst (Attachment 3). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Methods 3550B and 8270C

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Editions upon promulgation, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **approved May 2001**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **4**, 12/**10**/**03**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Quality Control SOP 13.16, "Top Loading Balance Calibration & Maintenance."

16.16 Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW846 and NYSASP"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 - Method Detection Limits

17.2 Attachment 2 - Extraction Worksheet

17.3 Attachment 3 – Single Analyst Capability Study

Attachment 1

Preparation Date: January 13, 2004			GCMS Method 3550B/8270C Soil Sonication Semivolatiles											
Analysis Date: January 14, 19, 2004						Using higher of three instrument MDLs								
Instrument: 5972hp60/64/66														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg
n-Nitrosodimethylamine	73.54	36.91	56.68	104.29	51.56	80.77	62.00	43.41	46.55	61.75	83.5	21.29	61.6	330
Pyridine	27.23	6.21	8.23	44.03	56.81	43.74	41.33	31.38	5.03	29.33	83.5	19.05	55.2	330
Benzaldehyde	37.45	61.98	82.18	51.16	69.25	58.32	55.08	86.02	26.74	58.69	166.5	19.24	55.7	330
Phenol	73.94	82.27	86.96	93.16	89.68	97.75	87.21	95.34	75.28	86.84	83.5	8.37	24.2	330
Bis(2-chloroethyl)ether	62.87	66.02	68.85	72.12	66.75	78.39	74.22	78.62	70.11	70.88	83.5	5.46	15.8	330
2-Chlorophenol	66.23	66.81	71.10	72.79	72.59	75.85	70.97	75.65	62.49	70.50	83.5	4.49	13.0	330
1,3-Dichlorobenzene	64.20	70.42	75.62	69.66	69.10	70.79	75.07	78.85	71.16	71.65	83.5	4.30	12.4	330
1,4-Dichlorobenzene	64.96	74.34	77.35	74.46	69.06	79.66	78.66	75.91	71.31	73.97	83.5	4.77	13.8	330
Benzyl alcohol	19.50	43.85	39.18	57.23	53.65	40.52	29.80	39.05	37.48	40.03	83.5	11.35	32.9	330
1,2-Dichlorobenzene	63.38	70.67	75.96	75.33	65.44	73.09	81.60	73.90	67.05	71.82	83.5	5.78	16.7	330
2-Methylphenol	54.43	67.63	74.78	83.93	82.13	76.71	67.76	74.58	58.62	71.17	83.5	10.00	29.0	330
2,2'-oxybis(1-Chloropropane)	56.97	59.97	63.06	61.97	68.85	72.86	64.51	71.07	60.49	64.42	83.5	5.40	15.7	330
Acetophenone	59.67	62.81	64.83	72.62	67.03	69.87	68.16	74.31	60.61	66.66	83.5	5.13	14.9	330
3-/4-Methylphenol	125.68	142.20	144.21	164.59	157.78	160.13	144.12	154.13	130.97	147.09	166.5	13.23	38.3	330
n-Nitroso-di-n-propylamine	60.94	62.89	77.11	68.54	62.65	80.12	81.32	71.36	57.86	69.20	83.5	8.76	25.4	330
Hexachloroethane	61.60	66.17	73.72	72.23	59.12	69.36	78.04	73.00	66.46	68.86	83.5	6.10	17.7	330
Nitrobenzene	88.31	105.32	102.21	113.21	103.54	103.45	95.20	115.00	91.72	102.00	83.5	9.01	26.1	330
Isophorone	71.44	75.80	82.66	91.87	84.34	88.08	80.08	93.51	73.74	82.39	83.5	7.85	22.7	330
2-Nitrophenol	51.75	61.16	72.06	68.23	69.17	71.70	63.38	75.27	55.94	65.41	83.5	7.92	22.9	330
2,4-Dimethylphenol	31.17	74.61	60.06	101.90	89.44	55.57	36.99	58.74	46.68	61.68	83.5	23.37	67.7	330
Bis(2-chloroethoxy)methane	61.62	71.47	71.46	80.74	75.31	75.30	73.16	80.41	66.62	72.90	83.5	6.13	17.7	330
2,4-Dichlorophenol	59.44	69.63	78.45	83.79	75.64	74.74	70.31	77.78	67.13	72.99	83.5	7.21	20.9	330
1,2,4-Trichlorobenzene	70.28	82.66	83.47	83.99	76.30	83.04	86.32	81.01	71.22	79.81	83.5	5.81	16.8	330
Naphthalene	62.57	75.46	77.48	77.25	70.85	75.46	77.32	72.12	66.44	72.77	83.5	5.31	15.4	330
4-Chloroaniline	51.13	107.88	106.36	131.95	124.51	111.02	60.50	106.91	99.04	99.92	166.5	27.02	78.3	330
Hexachlorobutadiene	75.40	74.95	79.96	85.87	79.30	90.72	76.91	90.30	75.81	81.02	83.5	6.33	18.3	330
Caprolactam	44.91	15.70	63.02	44.40	47.84	49.60	54.25	45.63	55.05	46.71	83.5	13.10	37.9	330
4-Chloro-3-methylphenol	78.00	78.72	94.01	99.18	97.95	83.18	83.05	95.61	78.24	87.55	83.5	8.98	26.0	330
2-Methylnaphthalene	71.62	73.41	78.65	83.14	81.57	83.86	80.57	87.77	72.51	79.23	83.5	5.64	16.3	330
1-Methylnaphthalene	77.50	79.53	88.83	92.05	91.04	91.16	85.07	93.01	75.77	86.00	83.5	6.76	19.6	330
Hexachlorocyclopentadiene	544.93	648.34	668.88	688.14	637.85	668.90	669.18	650.54	568.90	638.41	835	48.85	141.5	330
2,4,6-Trichlorophenol	56.28	56.66	61.12	71.59	72.14	66.28	65.79	68.88	52.43	63.46	83.5	7.15	20.7	330
2,4,5-Trichlorophenol	53.67	62.04	63.08	75.09	74.17	73.25	60.47	67.34	60.85	65.55	83.5	7.38	21.4	330
1,1'-Biphenyl	72.81	78.16	82.10	85.25	89.38	91.15	81.15	90.03	74.72	82.75	83.5	6.72	19.5	330

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 the reporting limit.

Attachment 1 (continued)

Preparation Date: January 13, 2004			GCMS Method 3550B/8270C Soil Sonication Semivolatiles											
Analysis Date: January 14, 19, 2004						Using higher of three instrument MDLs								
Instrument: 5972hp60/64/66														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg
2-Chloronaphthalene	74.71	75.64	86.21	90.74	90.46	88.67	82.97	88.79	77.04	83.91	83.5	6.54	18.9	330
2-Nitroaniline	58.02	65.00	71.12	72.14	76.42	72.84	68.38	78.00	63.37	69.48	83.5	6.44	18.6	670
Dimethylphthalate	54.98	64.82	67.72	78.14	68.80	66.99	70.31	65.25	58.38	66.15	83.5	6.70	19.4	330
2,6-Dinitrotoluene	59.61	64.33	76.00	83.89	81.42	73.18	74.99	73.70	66.03	72.57	83.5	7.94	23.0	330
Acenaphthylene	65.84	72.10	74.16	82.42	78.78	78.32	71.35	78.77	66.03	74.20	83.5	5.87	17.0	330
3-Nitroaniline	60.77	102.52	100.18	120.66	114.77	109.50	80.45	105.53	98.50	99.21	166.5	18.34	53.1	670
Acenaphthene	71.02	77.05	82.79	85.55	86.15	86.62	79.29	86.11	72.56	80.79	83.5	6.09	17.6	330
2,4-Dinitrophenol	212.71	327.15	360.93	384.16	380.38	380.85	366.65	361.80	325.15	344.42	835	53.95	156.2	670
4-Nitrophenol	178.78	210.41	239.96	282.02	260.89	225.23	222.65	247.16	215.20	231.37	334	30.29	87.7	670
2,4-Dinitrotoluene	51.19	58.75	59.21	75.40	75.44	69.99	63.14	67.46	57.37	64.22	83.5	8.42	24.4	330
Dibenzofuran	69.48	74.69	75.31	86.64	84.68	84.28	77.27	84.24	72.29	78.76	83.5	6.29	18.2	330
Diethylphthalate	72.32	80.45	83.98	95.71	96.08	89.06	83.29	88.78	75.36	85.00	83.5	8.27	23.9	330
4-Chlorophenyl-phenylether	61.65	68.02	75.24	82.91	75.06	75.61	76.42	73.51	63.32	72.42	83.5	6.81	19.7	330
Fluorene	57.92	67.69	71.79	80.99	74.69	72.61	74.54	68.57	64.00	70.31	83.5	6.73	19.5	330
4-Nitroaniline	89.86	106.49	122.11	153.03	146.46	145.06	113.59	126.30	114.29	124.13	166.5	20.84	60.4	670
4,6-Dinitro-2-methylphenol	56.37	59.15	65.64	95.18	79.79	72.98	65.12	80.29	57.75	70.25	166.5	12.94	37.5	670
n-Nitrosodiphenylamine	69.32	77.08	81.14	90.77	93.64	80.07	77.42	81.37	69.56	80.04	83.5	8.24	23.9	330
1,2-Diphenylhydrazine	72.81	77.24	84.73	94.84	98.72	88.31	88.59	93.47	79.56	86.47	83.5	8.66	25.1	330
4-Bromophenyl phenylether	55.10	66.68	70.72	77.22	72.96	73.75	67.68	65.41	60.21	67.75	83.5	6.94	20.1	330
Hexachlorobenzene	72.99	86.16	91.52	101.17	88.93	90.50	92.39	84.54	70.88	86.56	83.5	9.54	27.6	330
Atrazine	98.57	99.39	108.97	128.50	133.74	115.29	114.86	124.14	103.32	114.09	83.5	12.73	36.9	330
Pentachlorophenol	96.06	30.42	246.05	104.64	29.64	85.69	69.53	166.00	56.64	98.30	166.5	69.37	200.9	670
nanthrene	70.74	76.67	78.82	90.12	91.29	85.86	81.23	84.13	73.31	81.35	83.5	7.15	20.7	330
ıracene	68.11	75.98	80.28	93.27	92.43	82.30	76.26	83.05	71.99	80.41	83.5	8.51	24.6	330
Carbazole	132.41	143.63	149.48	165.17	168.87	163.06	153.24	157.84	142.56	152.92	83.5	12.03	34.8	330
Di-n-butylphthalate	71.10	78.99	79.60	87.68	95.89	86.74	82.34	84.07	72.76	82.13	83.5	7.67	22.2	330
Fluoranthene	61.50	77.99	76.59	87.08	81.46	77.17	78.18	73.57	65.26	75.42	83.5	7.84	22.7	330
Pyrene	93.60	93.74	106.35	114.61	115.20	103.69	99.93	103.47	90.19	102.31	83.5	8.94	25.9	330
Butylbenzylphthalate	79.48	79.01	88.72	98.38	102.10	91.73	88.51	87.45	80.00	88.38	83.5	8.20	23.7	330
3,3'-Dichlorobenzidine	39.23	53.97	53.19	66.94	62.45	56.55	45.02	52.90	47.90	53.13	83.5	8.49	24.6	670
bis(2-ethylhexyl)phthalate	94.66	90.23	96.69	100.05	115.74	103.37	99.39	102.32	88.84	99.03	83.5	8.03	23.3	330
Benzo(a)anthracene	66.88	83.59	82.54	91.66	88.81	84.86	86.07	80.79	71.01	81.80	83.5	8.04	23.3	330
Chrysene	84.92	94.28	98.99	106.15	112.18	101.54	90.42	98.56	87.66	97.19	83.5	8.84	25.6	330
Di-n-octylphthalate	66.73	67.90	71.98	75.80	79.63	79.07	68.65	72.07	62.66	71.61	83.5	5.75	16.6	330
Benzo(b)fluoranthene	67.60	69.92	82.53	85.85	80.98	80.41	77.59	82.57	68.96	77.38	83.5	6.80	19.7	330
Benzo(k)fluoranthene	74.44	83.99	79.29	101.04	102.70	83.25	85.97	87.20	74.17	85.78	83.5	10.22	29.6	330
Benzo(a)pyrene	68.96	74.72	75.32	87.80	88.48	74.91	71.02	76.17	68.11	76.17	83.5	7.37	21.3	330
Indeno(1,2,3-c,d)pyrene	63.83	66.04	68.62	80.57	78.27	69.95	69.81	74.03	59.63	70.08	83.5	6.70	19.4	330
Dibenzo(a,h)anthracene	64.32	71.46	71.79	81.88	81.42	74.01	68.47	75.17	63.74	72.47	83.5	6.50	18.8	330
Benzo(g,h,i)perylene	67.90	68.50	75.59	79.48	81.67	72.91	68.19	73.19	61.97	72.16	83.5	6.21	18.0	330

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 the reporting limit.

Attachment 2

EXTRACTION WORKSHEET

ASSIGNED TO: _____ **Low Level Soil S-V Method 3550B for 8270C**

CompuChem

DATE EXTRACTED/POSTED: **1/20/2004**

EMP ID NUMBER: _____ L176

BATCH NO. _____

COMPUCHEM NUMBER	CLIENT SAMPLE ID	QC SAMPLE TYPE	SAMPLE WEIGHT (g)	FINAL VOLUME (ml)	COMMENTS
					Add 1.0 mL of validation spike to LCS and SSs unless otherwise noted
					GPC (3640A) PERFORMED Y / N

SURROGATE	NO.	393	
	AMT.	0.5 ml	
	LOT		
SPIKE	NO.		VALID.
	AMT.		1.0 ml
	LOT		

Analyst initials. Extracted _____ KD _____ N2 _____ Bottle up _____

FINAL VOLUME VERIFIED: _____

SUPERVISOR REVIEWED: _____

SURROGATE & SPIKE ADDED BY: _____/_____
Initials Date

Witness _____/_____
Initials Date

Manufacturer and lot number of reagents/solvents used _____

Rev. 7/18/03.doc

Note: Attachment is subject to change without notice.

Attachment 3

Laboratory Name/North Carolina Certificate Number: CompuChem/79															
Analyst: Gracure Bailey															
Study Date: November 29, 2001															
Method: 3550B/8270C Soil															
Compounds	TrueVal	Rep#1	Rep#2	Rep#3	Rep#4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	-3SD	+3SD	SOP	RSD
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	%R	%R	%R	of (x)	of (x)	% R	%R	%RSD	%
Bis(2-chloroethyl)ether	2667	2249	1914	2012	2000	2044	77	19-113	143.63	1612.9	2474.6	79	121	50	7
2,2'-oxybis(1-Chloropropane)	2667	2393	1780	1689	1600	1866	70	18-114	90.00	1595.5	2135.5	86	114	50	5
N-Nitroso-di-N-propylamine	2667	2275	2001	1895	2800	2243	84	12-140	404.53	1029.2	3456.3	46	154	50	18
Hexachloroethane	2667	2104	2044	1534	1800	1871	70	10-131	260.02	1090.4	2650.6	58	142	50	14
Nitrobenzene	2667	2415	2175	2358	2200	2287	86	18-127	117.67	1934.0	2640.0	85	115	50	5
Isophorone	2667	2391	2182	1861	2000	2109	79	21-117	229.66	1419.5	2797.5	67	133	50	11
Bis(2-chloroethoxy)methane	2667	2340	1989	1938	1900	2042	77	22-141	202.15	1435.3	2648.2	70	130	50	10
Naphthalene	2667	2112	2181	1946	2400	2160	81	14-113	188.10	1595.5	2724.0	74	126	50	9
4-Chloroaniline	2667	1926	1680	1949	1800	1839	69	10-107	124.46	1465.4	2212.1	80	120	50	7
Hexachlorobutadiene	2667	2095	2359	1814	2300	2142	80	11-137	246.20	1403.4	2880.6	66	134	30	11
2-Methylnaphthalene	2667	2225	2266	1948	2100	2135	80	16-146	143.13	1705.4	2564.1	80	120	50	7
Hexachlorocyclopentadiene	2667	2739	2856	1960	1700	2314	87	10-150	570.58	602.0	4025.5	26	174	50	25
2-Chloronaphthalene	2667	2371	2438	2124	2300	2308	87	15-138	135.14	1902.8	2713.7	82	118	50	6
2-Nitroaniline	2667	2321	2163	1874	2300	2165	81	20-132	205.95	1546.7	2782.3	71	129	50	10
Dimethylphthalate	2667	2353	2419	1946	2500	2305	86	20-129	246.44	1565.2	3043.8	68	132	50	11
2,6-Dinitrotoluene	2667	2514	2475	2079	2500	2392	90	18-131	209.29	1764.1	3019.9	74	126	50	9
Acenaphthylene	2667	2245	2380	2018	2500	2286	86	18-121	206.67	1665.7	2905.8	73	127	50	9
3-Nitroaniline	2667	2194	1870	1745	2000	1952	73	11-105	191.87	1376.6	2527.9	71	129	50	10
Acenaphthene	2667	2110	1584	2046	2400	2035	76	18-112	337.82	1021.5	3048.5	50	150	30	17
2,4-Dinitrotoluene	2667	2399	2494	1636	2400	2232	84	18-128	399.99	1032.3	3432.2	46	154	50	18
Dibenzofuran	2667	2254	2409	2210	2300	2293	86	24-131	85.47	2036.8	2549.7	89	111	50	4
Diethylphthalate	2667	2272	2584	2251	2400	2377	89	18-128	153.06	1917.6	2835.9	81	119	50	6
4-Chlorophenyl-phenylether	2667	2159	2348	1554	2500	2140	80	18-130	414.98	895.3	3385.2	42	158	50	19
Fluorene	2667	2206	2288	2210	2500	2301	86	15-123	137.93	1887.2	2714.8	82	118	50	6
4-Nitroaniline	2667	2113	2109	2033	2500	2189	82	10-113	210.74	1556.5	2821.0	71	129	50	10
N-Nitrosodiphenylamine	2667	2419	2272	2667	2300	2415	91	23-123	179.99	1874.5	2954.5	78	122	30	7
4-Bromophenyl-phenylether	2667	2338	2540	2395	2600	2468	93	21-131	122.25	2101.5	2835.0	85	115	50	5
Hexachlorobenzene	2667	2538	2634	2102	2600	2469	93	15-148	247.54	1725.9	3211.1	70	130	50	10
Phenanthrene	2667	2279	2292	2144	2500	2304	86	18-123	146.95	1862.9	2744.6	81	119	50	6
Anthracene	2667	2308	2262	2462	2700	2433	91	19-120	197.48	1840.6	3025.4	76	124	50	8
Carbazole	2667	2386	2407	2723	3500	2754	103	24-140	520.68	1192.0	4316.0	43	157	50	19
Di-n-butylphthalate	2667	2439	2959	2511	2300	2552	96	20-131	284.96	1697.4	3407.1	67	133	50	11
Fluoranthene	2667	2336	2417	2155	3000	2477	93	16-127	365.47	1380.6	3573.4	56	144	30	15
Pyrene	2667	2584	2078	2228	2800	2423	91	12-123	329.19	1434.9	3410.1	59	141	50	14
Butylbenzylphthalate	2667	2668	2172	2103	2300	2311	87	20-119	251.76	1555.5	3066.0	67	133	50	11
3,3'-Dichlorobenzidine	2667	1587	1287	1470	1900	1561	59	10-129	257.52	788.4	2333.6	51	149	50	16
bis(2-ethylhexyl)Phthalate	2667	2712	3257	2132	2200	2575	97	18-125	523.06	1006.1	4144.4	39	161	50	20
Benzo(a)anthracene	2667	2336	2195	2026	2900	2364	89	17-117	378.98	1227.3	3501.2	52	148	50	16
Chrysene	2667	2316	2144	2135	2900	2374	89	19-121	360.58	1292.0	3455.5	54	146	50	15
Di-n-octylphthalate	2667	2457	2061	1725	2000	2061	77	13-135	301.90	1155.0	2966.5	56	144	30	15
Benzo(b)fluoranthene	2667	2137	2042	1892	2700	2193	82	10-126	352.89	1134.1	3251.4	52	148	50	16
Benzo(k)fluoranthene	2667	2081	2070	1518	2500	2042	77	12-129	402.76	834.0	3250.5	41	159	50	20
Benzo(a)pyrene	2667	1971	1985	1714	2700	2093	78	15-120	423.73	821.3	3363.7	39	161	30	20
Indeno(1,2,3-c,d)pyrene	2667	1664	1771	1539	3500	2119	79	15-127	925.87	-659.1	4896.1	-31	231	50	44
Dibenzo(a,h)anthracene	2667	1747	2002	1747	3000	2124	80	10-130	596.24	335.3	3912.7	16	184	50	28
Benzo(g,h,i)perylene	2667	1842	1857	1692	3200	2148	81	13-125	705.44	31.4	4264.1	1	199	50	33

Note: Attachment is subject to change without notice.

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-1022 SOP Section #: 2.5.2.2 Revision #: 11

SOP Title: Extraction of TCLP Leachate for Semivolatiles by SW-846 and NYSASP

Effective date: (QA fills in) 4/6/06

- Procedure prepared by: [signature] Date: 3/24/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 4/6/06
- Reason for change: Annual review & addition of DoD-ASM Requirements
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, 3510C, NYSASP, June 2000 and Revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -1022: Extraction of TCLP Leachate for Semivolatiles by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure -1022: Extraction of TCLP Leachate for Semivolatiles by SW-846 and NYSASP

1.0 Scope and Application

This procedure is designed to prepare water sample for GC/MS semivolatile analysis from the leachate generated using Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)". Acid and base/neutral fractions are extracted by separatory funnel, combined, dried, concentrated, and analyzed by GC/MS.

2.0 Summary

A measured volume of TCLP leachate, approximately 200 **mL**, is diluted to 1000 **mL** with extracted water (5x dilution) before solvent extraction using the separatory funnel technique. The two methylene chloride extracts (acid and base/neutral) fractions are combined, dried, concentrated, and bottled at a 1.0 **mL** final volume. Alternatively, acid/neutral and base fractions may be generated separately.

Method 3510C provides for either a base/neutral extraction at a pH > 11, followed by an acid extraction at a pH < 2 or, an initial acid/neutral extraction at a pH < 2, followed by a base extraction at a pH > 11. Currently, we perform an acid/neutral extraction followed by a base extraction (unless otherwise required by the client or project).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the **reporting limit** is not at least three times higher than the **calculated** MDL value, the **reporting limit** is adjusted upward in order to achieve this minimal ratio.

3.3 Reporting Units – μg/L

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD-QSM** and South Carolina Department of Health and Environmental control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for **DoD-QSM** and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.5 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in reconstructed ion chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Interferences by phthalate esters are common in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Interference from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature and diversity of the site being sampled.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Glassware

6.1.1 2000 **mL** separatory funnel with Teflon stopcock

6.1.2 Drying column

6.1.2.1 Chromatographic column, approximately 400 mm x 19 mm ID

6.1.2.2 Plug the drying column with a small plug of furnaced glass wool.

6.1.2.3 A glass funnel with a small plug of glass wool may be substituted for the drying column.

6.1.2.4 Furnaced anhydrous sodium sulfate will be used in the drying column or glass funnel to dry the solvent extract.

6.1.3 10 **mL** concentrator tube, graduated (Kontes K-570050-1025 or equivalent).

6.1.4 500 **mL** Kuderna-Danish (K-D) evaporative flask (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs, rubber bands, or blue Keck clip.

6.1.5 Snyder column, three-ball macro (Kontes K-503000-0121 or equivalent)

6.1.6 Graduated cylinders, 1000 **mL**

6.1.7 250 **mL** Erlenmeyer flask, glass.

6.2 Pyrex glass wool

6.2.1 Anneal in a 400° C oven for 2 - 4 hours before use.

6.3 Silicon carbide boiling chips, approximately 10/40 mesh.

6.3.1 Heat to 400° C for 30 minutes or Soxhlet extract with methylene chloride for 4 hours.

6.4 Water bath, heated with concentric ring cover, capable of temperature control (to ± 2° C).

6.4.2 The bath should be used in a hood.

6.5 Nitrogen evaporation device equipped with a water bath that can be maintained at 35 - 40° C. The N-Evap by Organomation Associated, Inc., South Berlin, MA (or equivalent) is suitable.

6.6 Wide range pH paper, 0 - 14 pH range

6.7 1.0 **mL** serological pipets

6.8 Pasteur pipets, glass, disposable

6.9 GC autosampler amber vials, 1.8 **mL**, glass with Teflon®-lined septa, and threaded screw cap

7.0 Reagents and Standards

All standards are prepared by the Organic Standards chemist. Details for the preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.) They are stored separate from samples at 4 ± 2°C in the reach in unit in the laboratory when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methylene chloride – pesticide grade or equivalent

7.3 Sodium sulfate, (ACS) granular, anhydrous, furnaced.

7.3.1 Anneal in 500° C oven for 2-4 hours in a shallow tray.

7.4 Sodium hydroxide solution – 10 N

7.5 Sulfuric acid - concentrated

7.6 Semivolatile surrogate spiking solution

7.6.1 The surrogate solution #393 contains the following compounds at the listed concentrations:

Acids	Concentration (µg/mL)
Phenol-d_6	200
2,4,6-Tribromophenol	200
2-Fluorophenol	200

Base Neutrals	Concentration (µg/mL)
Nitrobenzene-d_5	100
Terphenyl-d_{14}	100
2-Fluorobiphenol	100

7.7 Semivolatile spiking solution

7.7.1 The TCLP BNA spiking solution contains the following compounds at the listed concentrations:

Acids	Concentration (µg/mL)
o-Cresol	50
m-Cresol	50
p-Cresol	50
Pentachlorophenol	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Base Neutrals	Concentration (µg/mL)

Base Neutrals	Concentration (μg/mL)
1,4-Dichlorobenzene	**50**
2,4-Dinitrotoluene	**50**
Hexachlorobenzene	**50**
Hexachloroethane	**50**
Hexachlorobutadiene	**50**
Nitrobenzene	**50**
Pyridine	**50**

8.0 Sample Preservation **and** Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All TCLP leachates must be extracted within 7 days of leachate generation.

8.3 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler at 2-4.4^{o} C for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared, extracted, and analyzed with every extraction batch of up to 20 samples. For SC DHEC a blank is analyzed every 10 samples.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS; blanks spike, BS; matrix spike blank for NYSASP) must be prepared with each extraction batch of up to 20 samples. For SC DHEC the LCS is analyzed every 10 samples.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD), are prepared for each SDG.

9.4 Duplicates

9.4.1 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of SC DHEC. The duplicate frequency requirement is satisfied with the MS/MSD.

9.5 Contingency

9.5.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.5.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration and Standardization

N/A

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

The sample preparation technician must complete the extraction worksheet (Attachment 2) including the manufacturer and lot number of the reagents/solvents used. Any unused portions must be z'ed out. The laboratory supervisor reviews the completed worksheet for accuracy and completeness and then signs it. The worksheet accompanies the sample to the analytical laboratory.

11.1 Glassware Preparation

11.1.1 Prep (rinse) all glassware with methylene chloride before use, including the drying column containing sodium sulfate. Do not use any glassware that appears to be dirty or cracked.

11.2 Extraction

11.2.1 Thoroughly mix sample before aliquotting. Using a graduated cylinder, measure 200 **mL** of the TCLP leachate and dilute to 1000 **mL** with DI water. Transfer to a 2-liter separatory funnel. Pipet 1.0 **mL** of #393 surrogate standard solution into the separatory funnel and mix well.

11.2.1.1 Alternately, 100 **mL** of sample may be diluted to 500 **mL** with DI water with 0.5 **mL** surrogate and/or spike standard added. The final volume is also reduced by half.

11.2.2 For the MS/MSD, follow the step above, using one original sample. If one has not been designated by the client and identified on the worksheet, then select a representative sample from the batch. Add 1.0 **mL** of TCLP BNA spiking solution to each matrix spike. Add 1.0 **mL** of TCLP BNA spiking solution. Add 1.0 **mL** #393 to both matrix spikes.

11.2.3 Prepare a LCS (BS) by measuring 500 **mL** of extraction fluid and transferring to a 2 liter separatory funnel and diluting to a liter with DI water. Add 1.0 **mL** TCLP BNA solution and 1.0 **mL** #393 standard.

11.2.3.1 Alternatively, 250 **mL** of the extraction fluid may be diluted to 500 **mL** and surrogate, spike and final volume reduced by half.

11.2.4 To prepare the leachate blank, use 500 **mL** of the TCLP leachate blank and dilute to 1000 **mL** with DI water. Add 1.0 **mL** of surrogate solution #393.

11.2.4.1 Alternatively, 250 **mL** of the leachate blank may be diluted to 500 **mL** and surrogate and final volume reduced by half.

11.2.5 To prepare a method blank, use 1000 **mL** of DI water and add 1.0 **mL** of surrogate solution #393.

11.2.6 Check the pH of the sample with wide range pH paper, and add ~1 **mL** of concentrated sulfuric acid. Stopper the separatory funnel and shake for 20-30 seconds, venting the stopcock several times. Check the pH. If the pH value is 2 or less, note the pH on the extraction worksheet. If the pH is not 2 or less, continue to add 1 **mL** of acid, stopper, and shake until the pH reaches a value of 2 or less. The sample is now ready for the acid/neutral extraction.

11.2.7 Add 60 **mL** of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting into the hood to release excess pressure. Allow the organic layer to separate from the water phase for at least 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the organic solvent, use

mechanical techniques to complete the phase separation. The optimum technique depends on the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means. Drain the methylene chloride into a 250 **mL** Erlenmeyer flask.

11.2.8 Add a second 60 **mL** of methylene chloride to the separatory funnel and repeat the extraction, combining the acid/neutral extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.2.9 After completing the third acid/neutral extraction, pour the combined extracts through the drying column into the K-D apparatus. Rinse the Erlenmeyer flask with a small amount of methylene chloride and add the rinse to the drying column. Rinse the drying column with approximately 10 **mL** of methylene chloride and collect the rinseate in the K-D apparatus.

11.2.10 Slowly add a few **mL**s of 10 N sodium hydroxide to each separatory funnel. Stopper and shake for 20-30 seconds, opening the stopcock to vent the sample into the hood several times. Check the pH. If the pH is 11 or greater, note the pH on the extraction worksheet. If the pH is less than 11, continue to add base. Shake the sample and measure the pH until the pH is 11 or greater.

Note: The pH adjustment may be reversed, i.e., base first.

11.2.11 Add 60 **mL** of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for 2 minutes, with periodic venting into the hood to release excess pressure. Allow the organic layer to separate from the water phase for at least 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the organic solvent, use mechanical techniques to complete the phase separation. The best technique depends on the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means. Drain the methylene chloride into a 250 **mL** Erlenmeyer flask.

11.2.12 Add a second 60 **mL** volume of methylene chloride to the separatory funnel and repeat the extraction, combining the base extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.2.13 After completing the third acid extraction, pour the combined extracts through the drying column in the K-D apparatus. Rinse the Erlenmeyer flask with a small amount of methylene chloride and add the rinse to the drying column. Rinse the drying column with about 10 **mL** of methylene chloride and collect the rinseate in the K-D apparatus. An alternative to the

drying column would be a powder funnel containing glasswool and muffled Na_2SO_4.

11.2.14 Assemble a 500 **mL** K-D evaporative flask, 10 **mL** concentrator tube, and a three-ball macro Snyder column. Prep all glassware with methylene chloride.

11.2.15 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro Snyder column. Pre-wet the Snyder column by adding 1 **mL** of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90° C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the extract in 15-30 minutes. At the proper rate of distillation, the balls of the Snyder column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reached 4 **mL**, remove the K-D apparatus from the bath. Allow it to drain and cool.

11.3 Nitrogen concentration technique

11.3.1 Place the concentrator tube in the N-EVAP and evaporate the solvent volume to 1.0 **mL** using a gentle stream of clean, dry nitrogen. Rinse the internal wall of the concentrator tube with methylene chloride as the extract concentrates. Alternatively, microsnyder concentration may be used.

11.3.2 Quantitatively transfer the contents of the concentrator tube to a GC autosampler amber vial. The label must contain the CompuChem number, the procedure code (-1022), and the date extracted. Complete all paperwork correctly, verify that final volumes are correct, and obtain supervisor review of paperwork and extracts. Deliver the sample concentrates and paperwork to the designated area in the GC/MS laboratory.

11.3.3 The extract is now ready for analysis using Instrument Procedure 477/478, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW-846 and NYSASP".

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for the Evaluation of Solid Waste: "Physical/Chemical Methods," SW-846, Third Edition, 12/96, Method 3510C

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.4 QCSOP: Proper Documentation Procedures

16.5 QCSOP: Numerical Data Reduction

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, approved **June 2003**, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, **December 2005**, plus revisions.

16.11 CompuChem Quality Manual, Revision **7**, **Update 1,** 12/**13**/**05**, plus revisions

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)".

16.15 Instrument Procedure 477/478, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW-846 and NYSASP"

16.16 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 **– TCLP Waste Characterization Leachate Worksheet**

17.2 Attachment 2 – Extraction Worksheet

Attachment 1

CompuChem, a Division of Liberty Analytical Corp.

TCLP WASTE CHARACTERIZATION LEACHATE

Assigned to:_____ Method 1311 Date Extracted: 3/24/2006

Employee No.:_____ SPP -814 Batch No.:_____

COMPUCHEM NUMBER	CLIENT SAMPLE ID	SAMPLE TYPE	PRE-TEST pH VALUE START	PRE-TEST pH VALUE FINAL	EXTRACTION FLUID AND VOL. (mL) ADDED[1] 1	EXTRACTION FLUID AND VOL. (mL) ADDED[1] 2	PARTICLE REDUCT. DONE (Y/N)	SAMPLE WEIGHT (g)	FINAL LEACH. pH VALUE	FINAL VOLUME (ml)	PERCENT SOLID	COMMENTS

LOADED TUMBLER CALIB. CHECK (MUST BE 30 rpm ± 2 rpm)	
TUMBLER #	CALC. RPM
(COUNT rpm FOR 30 sec. AND MULTIPLY NUMBER BY 2 TO CALCULATE rpm)	

ROTATION TIME ONLY

Date/Time Started_____/_____

Date/Time Stopped_____/_____

Room Temp._____

Manufacturer and lot # of reagent used_____

Final Vol. Verified:_____

Reviewed By:_____

Ext. Fluid 1 pH_____ (4.93 ± 0.05)

Ext. Fluide 2 pH_____ (2.88 ± 0.05)

Enter volume (mL) of Extraction Fluid added into appropriate column, e.g., enter volume into column 1 if Ext. Fluid #1 is used. Ensure that the fluid volume to sample weight ration is 20:1.

Rev. 7/29/03:dce

Attachment **2**

COMPUCHEM
SEMI-VOLATILE WATER SW-846 METHOD 3510C
EXTRACTION WORKSHEET
TCLP WASTE CHARACTERIZATION

ASSIGNED TO: _____ DATE EXTRACTED/POSTED: 3/24/2006

EMP ID NUMBER: _____ -1022 BATCH NO.: _____

	COMPUCHEM NUMBER	CLIENT SAMPLE ID	QC SAMPLE TYPE	SAMPLE VOLUME (mL)	FINAL EXTRACT VOLUME	ADJUSTED PH A/N	ADJUSTED PH BASE	COMMENTS
1								USE 100 ml OF TCLP LEACHATE AND
2								DILUTE TO 500 ml WITH EXTRACTED
3								WATER FOR ALL SAMPLES. ADD 0.5 ml
4								TCLP B/N ACID SPIKE TO SS'S AND
5								BS'S. ADD 0.5 ml 8393 SURROGATE
6								TO ALL SAMPLES. FINAL VOL. = 0.5 ml.
7								
8								USE 250 ml VOLUME FOR
9								LEACHATE BLANK AND DILUTE TO 500 ml.
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
				S-VOL	SURROGATE & SPIKE ADDED BY			
		SURROGATE	NO.	393				FINAL VOLUME VERIFIED _____
			AMT.	0.5 ml	_____/_____			
			LOT		Initials Date			
		TCLP	NO.					SUPERVISOR REVIEWED _____
		B/N ACID	AMT.	0.5 ml	WITNESS _____/_____			
		SPIKE	LOT		Initials Date			

Analyst initial. Extracted _____ KD _____ N2_____ Bottle up _____

Manufacturer and lot number of reagents/solvents used _____

Rev. 7/21/03cks

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-079 SOP Section #: 2.5.2.1 Revision #: 11

SOP Title: Preparation of Water Samples for the Analysis of Low-Level Semivolatiles by SW-846 & NYSASP

Effective date: (QA fills in) 3/19/04

- Procedure prepared by: Diane C. Ellerine Date: 1/15/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Linda Carter Date: 3/2/04
- Reason for change: OH VAP Compliance ; Annual Review
- This procedure meets the requirements of the following approved method references: SW-846, Update III, 3rd Edition, Method 3510C and Method 8270C; NY State Analytical Services Protocol (NYSASP), June 2000, plus revisions

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

ORIGINAL

Annual Review—Signature: _____ Date: 2/10/05

Annual Review—Signature: _____ Date: 4/7/06

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -079: Preparation of Water Samples for the Analysis of Low Level Semivolatiles by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure -079: Preparation of Water Samples for the Analysis of Low Level Semivolatiles by SW-846 and NYSASP

1.0 Scope and Application

This procedure is described the extraction of semivolatile organic compounds from water samples prior to GC/MS analysis.

Method detection limits (MDL) and reporting limits are shown in Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A 1-liter volume of sample is spiked with the semivolatile surrogate solution, and acid and base/neutral fractions are extracted by separatory funnel with methylene chloride. The extracts are dried, combined, concentrated, and submitted for GC/MS analysis. Alternatively, acid/neutral and base fractions may be generated separately.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the

concentration range of the calibration curve and the ability to meet method linearity requirements.

The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/L

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers **(US ACE) and SC DHEC** do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **and 10% for SC DHEC**. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

4.0 Interferences

4.1 Method interferences may be caused by contaminants in reagents, solvents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free of interferences under the conditions of the analysis by preparing and analyzing laboratory reagent blanks.

4.2 Matrix interferences may be caused by contaminants that were inadvertently co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be done to determine the possible matrix interferences.

4.2.1 For sample extracts demonstrating matrix interferences, gel permeation cleanup procedure, Method 3640A, is an option.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 2-liter separatory funnel with ground-glass stopper and stopcock

6.2 1-liter graduated cylinder

6.3 Erlenmeyer Teflon flask

6.4 Drying column

6.5 Kuderna-Danish apparatus

6.5.1 Concentrator tube

6.5.2 K-D flask

6.6 Glass stirring rod (can be used for all samples)

6.7 Boiling chips (silicon carbide, furnaced at 800° C for 30 minutes)

6.8 Rubber bands

6.9 Nitrogen evaporation device (Organomation, or equivalent)

6.10 pH paper - wide range

6.11 Glass wool

6.11.1 Furnace at 400° C for 4 hours prior to use

6.12 Serological pipet - 1.0 ml with 1/100 ml graduations

6.13 Amber 2-ml vial with a Teflon-lined screw cap

7.0 Reagents & Standards

Refer to the Standards Preparation Logbook 22 F for details on preparation of standards used in this procedure. All standards used in this procedure are prepared in optima grade methylene chloride. Standards are stored separate from samples at 4 ± 2°C in the reach in unit in the laboratory when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Sodium hydroxide solution **10 N**

7.3 Sulfuric acid - concentrated

7.4 Solvents – pesticide residue analysis grade, or equivalent

7.4.1 Acetone

7.4.2 Methanol

7.4.3 Methylene chloride

7.5 Sodium sulfate (ACS) - powdered, anhydrous.

7.5.1 Purify by heating at 400° C for four hours in a shallow tray. Cool in a desiccator, and store in a glass bottle.

7.6 Surrogate Standard Spiking Solution

7.6.1 Base/neutral and acid surrogate #393 is used at a volume of 1.0 ml per 1000-ml sample.

7.7 Base/Neutral and Acid Matrix Standard Spiking Solution

7.7.1 The matrix standard spiking solution consists of base/neutral and acid compounds listed in Table 1.

7.7.2 The 8270 validation spike is used at a volume of 1.0 ml per liter sample or QC.

Table 1. Base/Neutral and Acid Compounds in Matrix Spiking Solution

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-**methylphenol**
----------	1,4-Dichlorobenzene
4-Nitrophenol	N-Nitroso-di-n-propylamine

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Aqueous samples must be extracted within 7 days of sampling.

8.3 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank is prepared with each extraction batch of up to 20 samples. **For SC DHEC, the method blank is performed at a frequency of 10% for water samples.**

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS, or blank spike, BS, or matrix spike blank for NYSASP) must be prepared with each extraction batch of up to 20 samples. **For SC DHEC, the LCS is performed at a frequency of 10% for water samples.**

9.3 **Matrix Spike/Matrix Spike Duplicate**

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared for each sample delivery group (SDG). **For SC DHEC, the MS/MSD are performed at a frequency of 10% for water samples.**

9.4 **Duplicates**

9.4.1 Duplicates, at a frequency of **10%**, are required when processing samples submitted to meet the regulatory requirements of **South** Carolina **DHEC**. The MS/MSD satisfy the duplicate requirement.

9.5 Contingency

9.5.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.5.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration & Standardization

NA

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Preparation of Equipment

11.1.1 Cover all work areas with plastic-backed, absorbent table covering, with the plastic side down.

11.1.2 Assemble the following glassware for each sample to be processed:

11.1.2.1 one 2-liter separatory funnel with ground-glass stopper and stopcock

11.1.2.2 one, 1-liter graduated cylinder

11.1.2.3 one Erlenmeyer Teflon flask

11.1.2.4 one drying column

11.1.2.5 one Kuderna-Danish apparatus (consists of a concentrator tube and a K-D flask)

11.1.2.6 one glass stirring rod

11.1.3 Rinse each of the items listed above with methylene chloride. Empty the methylene chloride into a solvent waste container.

11.1.4 Add a small plug of furnaced glass wool to each drying column, then add 1-2 inches of prepared sodium sulfate to each drying column.

11.1.5 Label each piece of glassware with the sample number.

11.2 Sample Preparation and Extraction

11.2.1 Using a 1 liter graduated cylinder measure 1 liter (nominal) of sample. Record the initial volume on the worksheet. (Attachment 2)

11.2.2 Pour the sample into its associated separatory funnel.

11.2.3 Add 1.0 ml of surrogate standard #393 to each production sample by using the serological pipet. It is important to add exactly 1.0 ml, since surrogate recoveries are used to judge the efficiency of the extraction. Record the surrogate standard ID number, the lot number, and volume added, on the extraction worksheet.

11.2.4 A method blank is prepared using 1000 ml DI water and spiking with 1.0 ml of surrogate standard #393.

11.2.5 Use 1000-ml sample volume for the matrix spikes and 1000 mL of DI water for the LCS. Add 1.0 ml of surrogate standard #393 and 1.0 ml of the 8270 validation spike standard to the LCS and MS/MSD. Record the standard ID numbers, the lot numbers, and volume added on the extraction worksheet.

11.2.6 Measure the initial pH, using wide range pH paper.

11.2.7 Slowly add concentrated sulfuric acid to each separatory funnel. Stopper the separatory funnel and shake for 20-30 seconds, venting the stopcock several times. Check the pH. If the value is 2 or less, note the pH on the extraction worksheet. If the pH is not 2 or less, continue to add 1 ml at a time of the acid, and shake until the pH has reached a value of 2 or less. The sample is now ready for the acid-neutral extraction.

11.2.8 Add 60 ml of methylene chloride to the appropriate 2-liter separatory funnel. Stopper each funnel and shake vigorously for 2 minutes. Be careful to vent the stopcock frequently under the hood, until the pressure equalizes.

11.2.9 Allow each separatory funnel to hang undisturbed for approximately 10 minutes, to allow the layers to separate. If an emulsion larger than two-thirds the size of the bottom layer (methylene chloride) forms, steps must be taken to break it up. Emulsions may be broken stirring, passing through the stopcock very slowly, or centrifugation. The method used is determined by the severity of the emulsion.

11.2.10 When two distinct layers are obtained, drain the lower layer (methylene chloride) into the Erlenmeyer flask. Close the stopcock when the water layer reaches the stopcock. The object is to collect all of the methylene chloride and none of the water.

11.2.11 Repeat steps 11.2.8 - 11.2.10 two more times.

11.2.12 Upon completion of the third extraction, collect all of the organic layer in the Erlenmeyer flask. Pour the total extract through the drying column into the K-D apparatus. Rinse the Erlenmeyer flask with a small amount of methylene chloride and add the rinse to the drying column. Rinse the drying column with approximately 20 ml of methylene chloride and collect the rinse in the K-D apparatus.

11.2.12.1 An alternative to the drying column would be a powder funnel containing glasswool and muffled Na_2SO_4.

11.2.13 Slowly add **10 N** sodium hydroxide to each separatory funnel. Stopper the separatory funnel and shake for 20-30 seconds, venting the stopcock under the hood several times. Check the pH. If the pH is now 11 or greater, note the pH on the extraction worksheet. If the pH is not 11 or greater, continue to add 1-ml volumes of sodium hydroxide. Shake and measure the pH until a value of 11 or greater is reached.

Note: The pH adjustment may be reversed if necessary, i.e. base first.

11.2.14 Once a pH of 11 or greater has been reached and the pH recorded, the sample is ready for the base extraction. Perform steps 11.2.8 – 11.2.10 that will result in a combined acid/neutral and base extract.

11.3 Extract Concentration

11.3.1 **Pour the combined extract in a KD apparatus.** Add 2-3 boiling chips to each K-D flask and attach a Snyder column. If the Snyder column is dry after the rinsing process, add 1-2 ml of methylene chloride to the top of the Snyder column.

11.3.2 Place the **KD apparatus** on a water bath set at 85-95° C. Adjust the vertical position of the apparatus and water temperature as required to concentrate the extract in 15-30 minutes.

11.3.3 Remove each K-D flask from the bath as soon as an apparent volume of 4 ml is reached and allow it to drain.

11.3.4 Remove the Snyder column and K-D flask from the concentrator tube and remember to label the concentrator tubes with the proper label. Place the concentrator tube in the rack and continue the concentration on the Organomation. Alternatively, microsynder concentration may be used.

11.3.5 Final concentration volume should be 1.0 ml for production samples and blanks. For sample spikes and blank spikes the volume should also be 1.0 ml.

11.3.6 Transfer the entire extract volume with a transfer pipet to an amber, 2-ml autosampler vial. Label the vial appropriately to indicate Prep Code,

CompuChem number, and **extraction** date. At the time of transfer, note the final volume on the Extraction worksheet.

11.3.7 Complete the paperwork, mark the volume on the vials, and forward both to the **GC/MS area for analysis following Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW-846 and NYSASP."**

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

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15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

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16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 12/96, Methods 3510C and 8270C

16.2 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions.

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **approved May 2001**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **4**, 12/1**0**/**03**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW-846 and NYSASP."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Method Detection Limits

17.2 Attachment 2 - Extraction Worksheet

17.3 Attachment 3 – Single Analyst Capability Study

Attachment 1

Preparation Date: January 13, 2004			GCMS Method 3510C/8270C (acid first separatory funnel extraction) Aqueous Semivolatiles												
Analysis Date: January 14, 19, 2004					Using higher of three instrument MDLs										
Instrument: 5972hp60/64/66															
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit	ReportLimit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	OH VAP ug/L
n-Nitrosodimethylamine	1.01	0.73	0.73	0.77	0.55	1.06	1.12	0.74	0.80	0.83	2.50	0.19	0.54	10	10
Pyridine	0.78	1.02	0.10	0.43	1.36	1.29	0.76	0.51	1.05	0.81	2.50	0.41	1.20	10	10
Benzaldehyde	3.93	3.17	4.00	3.63	3.70	4.27	3.72	3.85	2.64	3.66	5.00	0.49	1.41	10	10
Phenol	1.66	1.52	1.60	2.36	2.39	1.55	1.88	1.88	2.04	1.88	2.50	0.33	0.96	10	10
Bis(2-chloroethyl)ether	2.01	1.78	1.90	1.92	1.99	2.32	2.02	1.92	1.57	1.94	2.50	0.20	0.58	10	10
2-Chlorophenol	1.94	1.71	1.88	1.88	1.96	2.21	2.05	1.78	1.50	1.88	2.50	0.20	0.59	10	10
1,3-Dichlorobenzene	2.05	1.72	2.08	1.86	2.08	2.28	1.88	1.98	1.56	1.94	2.50	0.22	0.62	10	10
1,4-Dichlorobenzene	2.11	1.81	2.21	1.95	2.05	2.43	2.16	2.25	1.73	2.08	2.50	0.22	0.64	10	10
Benzyl alcohol	1.83	1.63	1.51	1.93	1.62	2.01	2.04	1.71	1.53	1.76	2.50	0.20	0.59	10	10
1,2-Dichlorobenzene	2.04	1.78	2.21	1.86	2.05	2.41	2.06	2.09	1.64	2.02	2.50	0.23	0.67	10	10
2-Methylphenol	1.99	1.41	1.82	1.81	1.74	2.21	2.01	1.78	1.40	1.80	2.50	0.27	0.77	10	10
2,2'-oxybis(1-Chloropropane)	2.06	1.63	1.99	1.93	1.90	2.31	2.06	1.88	*	1.97	2.50	0.19	0.58	10	10
Acetophenone	2.36	1.86	2.38	2.21	2.19	2.56	2.21	2.20	1.87	2.20	2.50	0.23	0.66	10	10
3-/4-Methylphenol	3.64	3.18	3.71	3.57	3.52	4.29	3.90	3.55	*	3.67	5.00	0.32	0.97	10	10
n-Nitroso-di-n-propylamine	1.79	1.13	1.56	1.60	1.46	1.84	1.55	1.71	1.23	1.54	2.50	0.24	0.69	10	10
Hexachloroethane	1.91	1.65	1.91	2.03	1.77	2.16	2.10	2.05	1.57	1.91	2.50	0.20	0.59	10	10
Nitrobenzene	3.38	3.74	3.14	3.80	3.09	3.73	3.58	3.12	3.63	3.47	2.50	0.29	0.84	10	10
Isophorone	2.45	2.03	2.30	2.34	2.23	2.54	2.19	2.34	1.91	2.26	2.50	0.20	0.57	10	10
2-Nitrophenol	1.85	1.64	1.70	1.87	1.57	2.13	1.91	1.63	1.68	1.78	2.50	0.18	0.52	10	10
2,4-Dimethylphenol	2.06	1.96	2.17	1.98	1.92	2.22	2.32	1.03	*	1.96	2.50	0.40	1.20	10	10
Bis(2-chloroethoxy)methane	2.23	1.85	2.08	2.11	2.03	2.57	2.23	2.15	1.95	2.13	2.50	0.21	0.59	10	10
2,4-Dichlorophenol	2.26	1.70	1.95	2.12	1.90	2.33	2.01	1.78	1.61	1.96	2.50	0.25	0.71	10	10
1,2,4-Trichlorobenzene	2.44	1.95	2.37	2.15	2.12	2.51	2.61	2.04	1.92	2.23	2.50	0.25	0.74	10	10
Naphthalene	2.42	2.03	2.30	2.30	2.36	2.73	2.51	2.25	2.04	2.33	2.50	0.22	0.63	10	10
4-Chloroaniline	4.27	3.31	4.00	4.13	4.00	4.49	4.01	3.65	2.60	3.83	5.00	0.57	1.66	10	10
Hexachlorobutadiene	2.37	1.93	2.28	2.21	1.99	2.49	2.56	2.34	1.89	2.23	2.50	0.24	0.71	10	10
Caprolactam	0.63	1.30	0.34	1.09	1.54	0.58	0.54	0.48	0.97	0.83	5.00	0.41	1.19	10	10
4-Chloro-3-methylphenol	2.55	2.09	2.31	2.59	2.27	2.91	2.73	2.50	2.22	2.46	2.50	0.26	0.76	10	10
2-Methylnaphthalene	2.50	1.98	2.26	2.30	2.21	2.58	2.15	2.25	1.93	2.24	2.50	0.21	0.61	10	10
1-Methylnaphthalene	2.65	2.25	2.62	2.52	2.52	2.89	2.54	2.37	2.22	2.51	2.50	0.21	0.60	10	10
Hexachlorocyclopentadiene	17.85	14.75	17.07	16.49	16.69	18.82	16.08	16.59	13.78	16.46	25.00	1.51	4.37	10	25
2,4,6-Trichlorophenol	2.18	1.66	1.92	1.83	1.81	2.11	1.80	1.97	1.60	1.88	5.00	0.19	0.55	10	10
2,4,5-Trichlorophenol	1.74	1.71	1.77	1.85	1.83	2.27	1.95	1.72	1.55	1.82	5.00	0.20	0.58	10	10
1,1'-Biphenyl	2.62	2.40	2.35	2.53	2.40	2.92	2.59	2.32	2.20	2.48	2.50	0.21	0.62	10	10

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 of the reporting limit.

Attachment 1 (continued)

Preparation Date: January 13, 2004			GCMS Method 3510C/8270C (acid first separatory funnel extraction) Aqueous Semivolatiles												
Analysis Date: January 14, 19, 2004						Using higher of three instrument MDLs									
Instrument: 5972hp60/64/66															
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	Std.Dev.	MDL	ReportLimit	ReportLimit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	OH VAP ug/L
2-Chloronaphthalene	2.51	2.17	2.46	2.47	2.50	2.83	2.63	2.29	2.20	2.45	2.50	0.21	0.61	10	10
2-Nitroaniline	1.62	1.39	1.51	1.63	1.63	1.71	1.41	1.60	1.20	1.52	2.50	0.16	0.47	20	20
Dimethylphthalate	2.09	1.75	1.98	1.98	2.05	2.29	1.96	2.09	1.82	2.00	2.50	0.16	0.46	10	10
2,6-Dinitrotoluene	2.15	1.85	2.01	2.05	2.05	2.34	1.96	1.96	1.70	2.01	2.50	0.18	0.52	10	10
Acenaphthylene	2.51	2.09	2.28	2.24	2.30	2.60	2.46	2.29	*	2.35	2.50	0.17	0.49	10	10
3-Nitroaniline	4.00	3.16	3.53	3.89	3.66	4.51	4.22	3.82	3.18	3.77	5.00	0.45	1.30	20	20
Acenaphthene	2.55	2.08	2.33	2.43	2.40	2.69	2.53	2.33	*	2.42	2.50	0.18	0.55	10	10
2,4-Dinitrophenol	7.03	4.63	7.58	6.83	6.73	8.53	6.38	7.55	5.47	6.75	25.00	1.16	3.37	20	25
4-Nitrophenol	7.25	8.60	8.38	9.19	10.78	6.67	7.21	7.43	8.84	8.26	10.00	1.28	3.69	20	20
2,4-Dinitrotoluene	2.12	1.72	2.02	1.99	2.08	2.34	1.96	2.12	1.80	2.02	2.50	0.18	0.53	10	10
Dibenzofuran	2.59	2.15	2.37	2.37	2.38	2.78	2.49	2.35	*	2.44	2.50	0.19	0.56	10	10
Diethylphthalate	2.09	1.78	1.97	2.02	2.13	2.32	1.92	2.06	1.78	2.01	2.50	0.17	0.50	10	10
4-Chlorophenyl-phenylether	2.23	1.84	2.15	2.17	2.21	2.47	2.04	2.20	1.85	2.13	2.50	0.20	0.57	10	10
Fluorene	2.64	2.17	2.36	2.41	2.42	2.77	2.55	2.41	*	2.47	2.50	0.18	0.55	10	10
4-Nitroaniline	4.25	3.54	4.00	3.94	4.03	4.79	4.46	3.52	*	4.07	5.00	0.43	1.30	20	20
4,6-Dinitro-2-methylphenol	2.62	2.20	2.92	2.73	3.14	3.25	3.27	2.69	*	2.85	5.00	0.37	1.10	20	20
n-Nitrosodiphenylamine	2.48	2.09	2.38	2.31	2.37	2.76	2.52	2.10	2.18	2.35	2.50	0.22	0.63	10	10
1,2-Diphenylhydrazine	2.40	1.91	2.23	2.33	2.33	2.62	2.20	2.30	1.99	2.26	2.50	0.21	0.61	10	10
4-Bromophenyl phenylether	2.33	2.05	2.13	2.20	2.26	2.67	2.37	2.20	*	2.28	2.50	0.19	0.57	10	10
Hexachlorobenzene	2.74	2.18	2.57	2.61	2.49	3.02	2.45	2.72	2.28	2.56	2.50	0.25	0.73	10	10
Atrazine	3.48	3.03	3.37	3.57	3.48	3.79	3.65	3.31	*	3.46	2.50	0.23	0.69	10	10
Pentachlorophenol	3.18	2.79	3.45	3.46	3.30	3.90	3.31	3.09	2.79	3.25	5.00	0.35	1.01	20	20
Phenanthrene	2.63	2.19	2.40	2.51	2.51	2.79	2.61	2.48	2.80	2.55	2.50	0.19	0.55	10	10
Anthracene	2.55	2.12	2.32	2.46	2.44	2.79	2.57	2.38	2.41	2.45	2.50	0.18	0.53	10	10
Carbazole	4.16	3.58	4.01	4.11	4.00	4.60	4.11	4.38	*	4.12	2.50	0.30	0.89	10	10
Di-n-butylphthalate	2.59	2.25	2.49	2.50	2.48	2.82	2.67	2.48	*	2.54	2.50	0.17	0.50	10	10
Fluoranthene	2.67	2.34	2.57	2.63	2.51	2.92	2.72	2.53	*	2.61	2.50	0.17	0.51	10	10
Benzidine	44.67	57.77	34.13	35.20	50.91	70.46	45.80	*	*	48.42	25.00	12.78	40.15	50	50
Pyrene	3.02	2.61	2.93	3.15	3.19	3.47	2.82	2.94	3.09	3.02	2.50	0.24	0.71	10	10
Butylbenzylphthalate	2.51	2.05	2.41	2.45	2.53	2.65	2.48	2.39	*	2.43	2.50	0.17	0.52	10	10
3,3'-Dichlorobenzidine	1.74	1.15	1.30	1.29	1.34	1.54	1.25	1.16	1.07	1.32	2.50	0.21	0.60	10	10
bis(2-ethylhexyl)phthalate	2.90	2.11	2.70	2.28	2.46	2.46	2.16	2.31	2.13	2.39	2.50	0.27	0.78	10	10
Benzo(a)anthracene	2.79	2.33	2.65	2.66	2.72	2.96	2.68	2.54	*	2.67	2.50	0.18	0.55	10	10
Chrysene	3.00	2.48	2.88	2.89	2.97	3.29	2.87	2.86	2.94	2.91	2.50	0.21	0.60	10	10
Di-n-octylphthalate	2.07	1.71	2.24	2.08	2.29	2.37	2.18	1.97	*	2.11	2.50	0.21	0.62	10	10
Benzo(b)fluoranthene	1.83	1.50	2.10	1.96	1.96	1.91	1.74	1.67	*	1.83	2.50	0.19	0.57	10	10
Benzo(k)fluoranthene	2.30	1.97	2.63	2.26	2.67	2.62	2.40	2.27	*	2.39	2.50	0.24	0.72	10	10
Benzo(a)pyrene	2.09	1.80	2.20	2.16	2.30	2.39	2.19	2.07	*	2.15	2.50	0.18	0.53	10	10
Indeno(1,2,3-c,d)pyrene	1.93	1.71	2.07	2.01	2.19	2.22	2.01	1.97	*	2.01	2.50	0.16	0.48	10	10
Dibenzo(a,h)anthracene	1.74	1.26	1.71	1.59	1.73	1.88	1.52	1.56	1.56	1.62	2.50	0.18	0.51	10	10
Benzo(g,h,i)perylene	2.30	2.01	2.30	2.41	2.53	2.63	2.39	2.25	*	2.35	2.50	0.19	0.56	10	10
* Data point eliminated using Dixon outlier test															

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 of the reporting limit.

Attachment 2

CompuChem
EXTRACTION WORKSHEET
SEMI-VOLATILE WATER
Method 3510C for 8270C

ASSIGNED TO: ______________________ DATE EXTRACTED/POSTED: 1/20/2004

EMP ID NUMBER: ______________________ BATCH NO.: ______________________

COMPUCHEM NUMBER	[illegible]	[illegible]	SAMPLE VOLUME (ml)	FINAL VOLUME (ml)	[illegible]	[illegible]	COMMENTS
							*Add 1.0 mL validation spike to LCS and SSs unless otherwise noted

		[illegible]	[illegible]
SURROGATE	NO.	393	
	AMT.	1.0 ml	
	LOT		
SPIKE	NO.		VALID.
	AMT.		1.0 ml
	LOT		

FINAL VOLUME VERIFIED: ______________________

SUPERVISOR REVIEWED: ______________________

SURROGATE & SPIKE ADDED BY: ________ / ________
Initials Date

Analyst initials. Extracted ________ KD ________ N2 ________ Bottle up ________

Witness ________ / ________
Initials Date

Manufacturer and lot number of reagents/solvents used ______________________

REV. 7/18/03 :doc

Note: Attachment is subject to change without notice.

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Attachment 3

Laboratory Name/North Carolina Certificate Number: CompuChem/79															
Analyst: Norma Bolton															
Study Date: November 21, 2001															
Method: 3510C/8270C & 625 Water															
Compounds	TrueVal	Rep#1	Rep#2	Rep#3	Rep#4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	-3SD	+3SD	SOP	RSD
	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l	%R	%R	%R	of (x)	of (x)	% R	%R	%RSD	%
Benzaldehyde	80	48.5	41.8	56.9	42.5	47	59	10-100	6.98	26.5	68.4	56	144	50	15
Phenol	80	38.8	36.9	38.8	34.1	37	46	15-100	2.23	30.4	43.8	82	118	30	6
Bis(2-chloroethyl)ether	80	69.3	55.7	56.2	62.0	61	76	35-100	6.32	41.8	79.8	69	131	50	10
2-Chlorophenol	80	76.4	75.0	79.5	65.3	74	93	39-102	6.12	55.7	92.4	75	125	50	8
2-Methylphenol	80	67.7	63.1	61.7	61.5	63	79	36-100	2.87	54.9	72.1	86	114	50	5
2,2'-oxybis(1-Chloropropane)	80	70.1	44.6	48.3	60.2	56	70	32-100	11.65	20.8	90.7	37	163	50	21
Acetophenone	80	71.0	68.6	72.2	57.6	67	84	28-100	6.68	47.3	87.4	70	130	50	10
3-, 4-Methylphenol	160	122	103	104.3	105.4	109	68	10-150	9.01	81.7	135.7	75	125	50	8
N-Nitroso-di-N-propylamine	80	76.2	57.6	63.8	56.2	59	74	23-121	4.05	47.1	71.3	79	121	50	7
Hexachloroethane	80	52.2	60.3	58.0	53.8	56	70	27-101	3.73	44.9	67.3	80	120	50	7
Nitrobenzene	80	64.4	62.6	63.3	71.9	66	82	35-103	4.28	52.7	78.4	80	120	50	7
Isophorone	80	63.7	65.9	69.5	66.9	66	83	37-101	2.40	59.3	73.7	89	111	50	4
2-Nitrophenol	80	87.4	95.3	95.0	56.3	84	104	39-104	18.51	28.0	139.0	34	166	30	22
2,4-Dimethylphenol	80	67.5	74.1	29.6	45.5	54	68	13-120	20.47	-7.3	115.6	-13	213	50	38
Bis(2-chloroethoxy)methane	80	75.2	62.7	69.4	71.3	70	87	41-112	5.19	54.0	85.2	78	122	50	7
2,4-Dichlorophenol	80	89.3	99.1	97.0	56.6	85	107	41-112	19.73	26.3	144.7	31	169	30	23
Naphthalene	80	85.0	71.8	69.5	52.4	70	87	30-100	13.40	29.5	109.9	42	158	50	19
4-Chloroaniline	80	83.0	60.4	72.4	61.5	69	87	33-102	10.59	37.6	101.1	54	146	50	15
Hexachlorobutadiene	80	60.3	83.7	75.2	69.7	72	90	29-107	9.80	42.8	101.6	59	141	30	14
Caprolactam	80	26.1	23.4	25.9	18.9	24	29	10-100	3.34	13.5	33.6	57	143	50	14
4-Chloro-3-methylphenol	80	88.1	89.3	94.9	66.9	85	106	45-110	12.30	47.9	121.7	57	143	30	14
2-Methylnaphthalene	80	78.7	68.0	71.8	56.5	69	86	30-130	9.27	41.0	96.6	60	140	50	13
Hexachlorocyclopentadiene	80	56.7	81.0	71.8	93.3	76	95	10-128	15.40	29.5	121.9	39	161	50	20
2,4,6-Trichlorophenol	80	94.0	104.2	106.8	65.5	93	116	41-126	18.92	35.9	149.4	39	161	30	20
2,4,5-Trichlorophenol	80	81.3	91.0	90.6	79.5	86	107	43-122	6.07	67.4	103.8	79	121	50	7
1,1'-Biphenyl	80	77.2	74.1	76.4	54.9	71	88	41-100	10.60	38.9	102.5	55	145	50	15
2-Chloronaphthalene	80	65.0	74.1	74.3	87.1	75	94	29-119	9.06	47.9	102.3	64	136	50	12
2-Nitroaniline	80	76.5	62.6	67.8	75.7	71	88	48-109	6.63	50.7	90.5	72	128	50	9

Note: Attachment is subject to change without notice.

Attachment 3 (continued)

Laboratory Name/North Carolina Certificate Number: CompuChem/79															
Analyst: Norma Bolton															
Study Date: November 21, 2001															
Method: 3510C/8270C & 625 Water															
Compounds	TrueVal	Rep#1	Rep#2	Rep#3	Rep#4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	-3SD	+3SD	SOP	RSD
	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l	%R	%R	%R	of (x)	of (x)	% R	%R	%RSD	%
Dimethylphthalate	80	79.5	77.6	79.8	72.3	77	97	44-100	3.49	66.8	87.8	86	114	50	5
2,6-Dinitrotoluene	80	67.6	75.0	80.0	79.9	76	95	46-117	5.85	58.1	93.2	77	123	50	8
Acenaphthylene	80	88.5	81.3	82.9	81.2	83	104	40-103	3.47	73.1	93.9	88	112	50	4
3-Nitroaniline	80	72.6	59.4	64.1	69.5	66	83	39-104	5.85	48.9	83.9	74	126	50	9
Acenaphthene	80	88.6	57.3	65.2	74.5	71	89	33-100	13.46	31.0	111.8	43	157	30	19
2,4-Dinitrophenol	80	28.8	16.1	85.5	88.1	55	68	10-117	37.51	-57.9	167.2	-106	306	50	69
4-Nitrophenol	80	33.2	45.3	44.4	52.9	44	55	10-100	8.14	19.5	68.4	44	156	50	19
2,4-Dinitrotoluene	80	65.5	76.4	80.1	99.2	80	100	36-116	14.02	38.2	122.4	48	152	50	17
Dibenzofuran	80	78.8	73.6	75.7	73.8	75	94	51-103	2.41	68.2	82.7	90	110	50	3
Diethylphthalate	80	79.0	74.3	80.1	65.6	75	93	44-103	6.62	54.9	94.6	73	127	50	9
4-Chlorophenyl-phenylether	80	79.6	78.1	80.2	84.2	81	101	43-107	2.62	72.7	88.4	90	110	50	3
Fluorene	80	91.6	85.9	90.7	71.2	85	106	36-106	9.42	56.6	113.1	67	133	50	11
4-Nitroaniline	80	83.4	62.8	60.6	73.5	70	88	26-108	10.49	38.6	101.5	55	145	50	15
4,6-Dinitro-2-methylphenol	80	72.5	62.7	84.8	59.4	70	87	10-130	11.42	35.6	104.1	51	149	50	16
N-Nitrosodiphenylamine	80	72.7	67.6	74.9	39.8	64	80	44-108	16.30	14.9	112.7	23	177	30	26
4-Bromophenyl-phenylether	80	75.6	77.6	83.3	51.6	72	90	48-108	13.98	30.1	114.0	42	158	50	19
Hexachlorobenzene	80	65	83.1	87.6	65.6	75	94	42-123	11.75	40.0	110.5	53	147	50	16
Atrazine	80	234.1	83	91	88.9	124	155	70-150	73.40	-96.1	344.3	-77	277	50	59
Pentachlorophenol	80	50.1	86.8	100.5	63.4	75	94	12-150	22.68	7.2	143.2	10	190	30	30
Phenanthrene	80	88.3	79.5	87.9	50.2	76	96	38-108	17.98	22.5	130.4	29	171	50	24
Anthracene	80	88.9	79.9	86.8	58.4	78	98	54-111	13.95	36.7	120.3	47	153	50	18
Carbazole	80	78.9	80.7	90.8	53.9	76	95	38-107	15.68	29.0	123.1	38	162	50	21
Di-n-butylphthalate	80	74.8	70.8	80.7	56.9	71	88	46-111	10.11	40.5	101.1	57	143	50	14
Fluoranthene	80	86.54	84.5	93.7	53.8	80	100	36-111	17.67	26.6	132.6	33	167	30	22
Pyrene	80	85.7	73.6	80.5	51.7	73	91	10-150	14.98	27.9	117.8	38	162	50	21
Butylbenzylphthalate	80	73	63.0	71.2	49.5	64	80	38-111	10.59	32.3	95.8	50	150	50	17
3,3'-Dichlorobenzidine	80	53.4	54	50	41.8	50	62	36-114	5.70	32.9	67.1	66	134	50	11
bis(2-ethylhexyl)Phthalate	80	72.5	62.6	73.0	51.2	65	81	20-107	10.25	34.1	95.6	53	147	50	16
Benzo(a)anthracene	80	89.3	76.7	84.0	54.2	76	95	35-106	15.47	29.6	122.4	39	161	50	20
Chrysene	80	88.5	77.1	85.0	55.1	76	96	37-110	14.98	31.5	121.4	41	159	50	20
Di-n-octylphthalate	80	60.0	62	72.4	61.8	64	80	40-117	5.65	47.1	81.0	74	126	30	9
Benzo(b)fluoranthene	80	68.4	80	84.3	71.3	76	95	33-112	7.32	53.9	97.8	71	129	50	10
Benzo(k)fluoranthene	80	76.2	77.0	87.7	47.3	72	90	37-109	17.31	20.1	124.0	28	172	50	24
Benzo(a)pyrene	80	72.6	77.2	83.6	60.8	74	92	37-106	9.61	44.7	102.4	61	139	30	13
Indeno(1,2,3-c,d)pyrene	80	64.9	73.5	81.0	66.7	72	89	39-104	7.30	49.6	93.4	69	131	50	10
Dibenzo(a,h)anthracene	80	72.8	77.9	86.2	64.8	75	94	31-109	8.99	48.5	102.4	64	136	50	12
Benzo(g,h,i)perylene	80	73.0	78.4	86.6	62.3	75	94	35-107	10.22	44.4	105.7	59	141	50	14

Note: Attachment is subject to change without notice.

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 194 SOP Section #: 2.2.5.3 Revision #: 6

SOP Title: GC/ECD Analysis of Polychlorinated Biphenyls (PCBs) as Aroclors in Water and Soil Extracts by SW-846 and NYSASP

Effective date: (QA fills in) 3/30/06

- Procedure prepared by: Meppadr Date: 3/30/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Kerry L. Hinshaw Date: 3/29/06
- Reason for change: Addition of DoD-QSM Requirements
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, Method 8082; New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date:

Instrument Procedure 194: GC/ECD Analysis of Polychlorinated Biphenyls (PCBs) as Aroclors in Water and Soil Extracts by SW-846 and NYSASP

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Instrument Procedure 194: GC/ECD Analysis of Polychlorinated Biphenyls (PCBs) as Aroclors in Water and Soil Extracts by SW-846 and NYSASP

1.0 Scope and Application

Method 8082 is used to determine the concentration of various polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid and liquid matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD). While Method 8082 also provides for the analysis of individual PCB congeners, this procedure only deals with the determination of PCBs as Aroclors. The following are amenable to analysis using this method:

Compound Name	CAS Registry No.
Aroclor-1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Prior to analysis the sample must be extracted following Sample Preparation Procedure – 1049, "PCBs in Water Preparation Procedure," Sample Preparation Procedure –735 (-233→945), "Low Level Preparation for Analysis of PCBs only in Soil/Sediment/Sludge". Sample Preparation Procedure –247, Sample Preparation Procedure –236, "Soxhlet Extraction of S/S/S Samples by Method 3540C in SW-846 & NYSASP,"or "Automated Soxhlet Extraction of Soil/Sediment/Sludge and Wipe

Samples by SW-846 + NYSASP." A variety of optional cleanup steps may be used, including Florisil, Gel Permeation Chromatography (GPC) or acid wash.

The analysis is performed using the dual column option described in Method 8082 where a Y-splitter joins a single injection port with two dissimilar analytical columns. In this manner, confirmation may be performed simultaneously with the primary analysis. After cleanup, the extract is analyzed by injecting 2 or 4 μL (1 or 2 μL per column) into a GC with dual wide-bore, fused silica, capillary columns with dual electron capture detectors (GC/ECD).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL **are** reported and qualified as estimated concentration**s**.

To meet the requirements of the DoD-QSM, the reporting limit must be at least three times higher than the **calculated** MDL value**. T**he **reporting limit may be** adjusted upward in order to achieve this minimal ratio.

3.3 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD-QSM** and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **for the DoD-QSM** and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.4 SC DHEC – South Carolina Department of Health and Environmental Control

3.5 Aroclor 1660 – Term to denote a mixture of Aroclor 1016 and 1260

3.6 DoD-QSM – Department of Defense-Quality Systems Manual

4.0 Interferences

4.1 Interferences in this method can be grouped into three broad categories: contaminated solvents, reagents, or glassware; contaminated GC carrier gas, parts, column surfaces, detector surfaces; and the presence of co-eluting compounds in the sample matrix that also are detected by the ECD. Specific cleanups may be necessary for samples, depending on the compounds of interest.

4.2 Interferences by phthalate esters introduced during the sample preparation procedures can be a major problem. Common flexible plastics found in certain gloves and other objects contain varying amounts of phthalates which may be introduced during lab operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent wetted surfaces are handled. These interferences can be minimized by cleanup of solvents, reagents, and glassware.

4.3 Glassware must be scrupulously cleaned as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130^{o} C for several hours or rinse with methanol and drain.

4.4 The presence of elemental sulfur will result in broad peaks that interfere with early eluting pesticides, and can be removed by cleaning the extract with shiny copper or by the use of GPC cleanup, or with tetrabutylammonium (TBA) sulfite reagent

4.5 Waxes, lipids, and other high molecular weight compounds are also removed using GPC.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Gas chromatograph: Varian 3400 and Trace 2000 suitable for splitless injection with all required accessories, including syringes, analytical columns, gases, ECDs, and integrator or data system.

6.2 The following wide-bore columns are used in the analysis:

6.2.1 Column 1-30 m x 0.53 mm ID fused silica capillary column bonded with 35% phenyl methylpolysiloxane (CLPesticides), 0.50 μm film thickness

6.2.2 Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50% phenyl methylpolysiloxane (CLPesticides2), 0.42 μm film thickness

6.2.3 Wide-bore columns are installed in a 1/4 inch injectors, with deactivated liners designed specifically for use with mega-bore columns

6.2.4 Restek Y-shaped fused silica connector

7.0 Reagents & Standards

Refer to the Standards Preparation Logbook 22 F for details on preparation of standards used in this procedure.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Reagent or pesticide grade chemicals must be used for all tests.

7.3 All standard solutions should be stored at 4^{o} C in Teflon-sealed containers in the dark. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if QC indicates a problem.

7.4 Stock standard solutions (1 mg/mL) are purchased from Restek Corporation, for example, as certified solutions, or they may be prepared from neat material.

7.5 Calibration standards are prepared at five concentrations by diluting the stock standards solutions with hexane.

7.6 Separate calibration standards are used for each multi-component analyte, with the exception of Aroclors 1016 and 1260, which can be run as a mixture. The mixture is referred to as Aroclor 1660.

7.6.1 The second source Initial Calibration Verification standard is Aroclor 1660 prepared at 100 ηg/mL.

7.7 Surrogate standards are added to all samples, method blanks, matrix spikes, laboratory control samples and calibration standards.

8.0 Sample Preservation **and** Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be analyzed within 40 days of extraction.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2 - 4^{o} C in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 **Routine Instrument Maintenance**

9.1.1 When linearity is difficult to achieve, verify that the appropriate length of column is inserted in the detector. Examine all column ends and determine if the ends of the columns need to be trimmed. The removal of 0.5 meters of the detector end of the column may be indicated when linearity is difficult to achieve.

9.1.2 When the instrument blanks fail, examine the chromatography to determine if there is contamination in the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the contamination is such that baking for 1 hour does not improve the baseline, it may be necessary to change the liner.

9.1.3 When calibration verifications fail recovery low, verify that the correct peaks are being named. Also verify that the syringe is not plugged and change the septa. If the calibration verification fails again, examine the chromatography to determine if there is a problem with the baseline that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. Also, if the calibration verification is failing on only one column, the y-splitter may be plugged. If the calibration verification continues to fail, the instrument will need a new calibration curve.

9.1.4 When calibration verifications fail recovery high, verify that the correct peaks are being named. Examine the chromatography to determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the calibration verification fails again, the instrument will need a new calibration curve.

9.1.5 When the calibration verification fails due to drift, change the septa and verify that all the column fittings are secure. Also, determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the calibration verification still fails, it is permissible to update retention times once per 24 hours period. Record in the instrument run log when the retention times are updated.

9.1.6 All preventive and routine maintenance as mentioned above is recorded in the instrument run log. Major maintenance is recorded in the maintenance log (Attachment 2).

9.2 Initial Calibration

9.2.1 The percent Relative Standard Deviation (%RSD) of the calibration factors for each selected Aroclor component in the initial calibration must be less than or equal to 20%. The % RSD for Aroclors is calculated by determining the mean of the average of the five selected peaks in each standard level. Acceptance criteria must be met by the average of the five peaks, as opposed to each individual peak.

9.2.2 When the %RSD is ≤ 20%, the instrument response is considered to be linear and the mean calibration factor can be used for quantitation. If the %RSD is greater than 20%, the analyst will investigate and correct the problem and repeat the initial calibration. Alternatively, a calibration

model, such as a least squares regression, is available in the ThruPut system.

9.2.2.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, the use of a quadratic fit to demonstrate the linearity of the initial calibration is not allowed. The use of a linear regression is permitted.

Note: The DoD-QSM does not allow the use of quadratic fit linear regression model.

9.2.2.2 When **the linear** option is used, the line must not be forced through the origin (zero) and the correlation coefficient of the equation must be 0.99 or greater for a valid calibration. **The DoD-QSM requires the correlation coefficient to be ≥ 0.995.**

9.3 Initial Calibration Verification

9.3.1 Analyze a second source initial calibration verification (ICV) standard to verify the standard curve before proceeding with analyses.

9.3.2 The acceptance criteria for the ICV is +/-20% of the true value.

9.4 Continuing Calibration

9.4.1 The calibration factors in all continuing calibration verification standards must be within ± 15% difference (%D) when compared to the mean calibration factors from the initial calibration on both analytical columns. The % D is calculated by determining the average %D of the five selected peaks for each Aroclor. Acceptance criteria must be met by the average of the five peaks, as opposed to each individual peak.

9.4.2 If the %D exceeds ± 15%, then corrective action must be taken and the continuing calibration verification re-injected. If the calibration still exceeds the criteria, a new initial calibration must be analyzed.

9.4.3 If any of the continuing calibration Aroclor components fall outside their retention time windows, the system is out of control and corrective action must be taken to correct the problem. If, after re-injection of the standard following corrective action, the retention times are still outside the windows, a new initial calibration must be analyzed.

9.5 Surrogates

9.5.1 Statistical control limits for surrogates are shown in the following table. Surrogate recoveries must be met for all samples, blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Surrogate	Percent Recovery Range	
	Water	Soil
Decachlorobiphenyl	43-144	43-144
Tetrachloro-m-xylene	43-135	43-135

If both surrogates (tetrachloro-m-xylene and decachlorobiphenyl) are outside the control range on both columns, the sample must be re-extracted and re-analyzed. Only one surrogate is required to pass on one column.

9.5.2 An exception to the above criteria is allowed for field samples and matrix spikes if the recovery is out of range due to interfering peaks. Chromatograms must be examined to determine if the out of control condition may be attributed to sample interferences, or to a partial injection, before re-extracting the sample.

9.5.2.1 Partial injections should be re-injected and can be diagnosed by comparing the solvent peak of the sample to the solvent peak of the standard. They should be similar in size.

9.5.3 The following table contains the DoD-QSM requirement for surrogate control limits. Surrogate recovery criteria must be met on both columns.

Surrogate	Aqueous	Solid
Decachlorobiphenyl	**40-135**	**60-125**

9.5.3.1 If the criteria in Section 9.5.3 are not met, the DoD-QSM requires the field and QC samples be reanalyzed or re-extracted (if sufficient sample remains).

9.5.3.2 Field and QC samples reported wit failing surrogates are qualified in the narrative, referencing the applicable DoD-QSM data flag (Q or J).

9.6 Method blanks

9.6.1 Method blanks are extracted with each preparation batch of up **to** 20 samples. For SC DHEC, the method blank is performed at a frequency of 10%.

9.6.2 The acceptance criteria for method blanks includes the following:

9.6.2.1 Surrogates acceptance criteria must be met.

9.6.2.2 No target analytes may be present above the reporting limit.

9.6.2.3 To meet the requirements of the DoD-QSM, no target analytes may be present at concentrations above half the reporting limit.

9.6.3 If the method blank exceeds acceptance criteria, the source of the problem (s) must be investigated and appropriate corrective action taken. All samples processed with a method blank that does not meet acceptance criteria must be re-extracted and reanalyzed.

9.7 Laboratory Control Sample (LCS)

9.7.1 A**n** LCS is DI water, or furnaced Ottawa sand or sodium sulfate for solid matrices, spiked with a target Aroclor and surrogates. The **LCS** is prepared with each preparation batch of samples. For SC DHEC, the **LCS** is performed at a frequency of 10%.

9.7.2 LCS recovery limits are as follows.

Analyte	Aqueous % Recovery	Solid % Recovery

Aroclor 1260	59-120	37-137
Aroclor 1016	63-132	50-145

9.7.2.1 The LCS control limits required by the DoD-QSM are as follows:

Analyte	Aqueous % Recovery	Solid % Recovery
Aroclor 1260	**25-145**	**40-140**
Aroclor 1016	**30-145**	**60-130**

9.7.2.2 If the LCS fails the DoD-QSM acceptance criteria, correct the problem and re-extract and reanalyze the associated samples.

9.7.2.3 If corrective actions fail, e.g., insufficient sample remains for extraction, report data associated with failing LCS. Qualify the specific analytes in the associated field and QC samples in the narrative, referencing the appropriate DoD-QSM data flag (Q).

9.7.3 Surrogate acceptance criteria must be met.

9.7.4 If the LCS fails to meet acceptance criteria, all samples in the associated batch must be re-extracted and reanalyzed.

9.8 Matrix Spikes

9.8.1 A matrix spike and matrix spike duplicate (MS/MSD) must be performed for each sample delivery group of up to 20 field samples. For SC DHEC, the matrix spike/matrix spike duplicate are performed at a frequency of 10% for water samples and 5% for soils.

9.8.2 The percent recovery and relative percent difference (RPD) criteria for the MS/MSD are shown in the following table.

Analyte	Aqueous % Recovery	Aqueous RPD	Solid % Recovery	Solid RPD
Aroclor 1660	50-150	20	50-150	20

9.8.2.1 To meet the requirements of the DoD-QSM, the duplicate matrix spike should meet the control limit requirements for the LCS. The %RPD should be ≤ 30%.

9.8.2.1.1 If the DoD-QSM acceptance criteria are not met for the duplicate matrix spikes, contact the client for guidance.

9.8.2.1.1 Qualify the original sample associated with the failing duplicate matrix spikes in the narrative. Reference DoD-QSM data qualifying flag (J).

9.8.3 The recoverability of spiked analytes in environmental samples is highly influenced by the particular matrix. When recoveries do not meet the acceptance criteria or interferences preclude proper assessment of the data, results of a**n** LCS are evaluated to verify that the analytical systems (sample preparation and analysis) are in control.

If the original unspiked sample does not meet surrogate acceptance criteria and the MS/MSD do meet criteria, the sample should be reanalyzed or re-extracted then reanalyzed. If the original sample and the MS/MSD yield the same unacceptable surrogate recoveries, then further action is not required since matrix interference would be confirmed.

9.9 Duplicates

9.9.1 Sample duplicates are required at a frequency of 10% for water samples and 5% for soil samples when processing samples submitted to meet the regulatory requirements of South Carolina DHEC. This requirement can be met with the MS/MSD.

9.10 Contingency

9.10.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.10.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.10.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.10.4 Any other issues that potentially affect data quality should also be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 Initial Calibration

10.1.1 For the initial calibration five concentration levels are prepared for Aroclors 1016/1260. Single standards for the remaining Aroclors are prepared at the mid-concentration. The M3 level for Aroclors 1221, 1232, 1242, 1248, and 1254 are used for pattern recognition and to generate calibration factors for them.

On a project-specific basis, three or five concentration levels may be required for the remaining Aroclors. The following table presents the concentrations in μg/mL of Aroclor standards if five levels are required. Each multi-component standard contains the two surrogates at the indicated levels.

Analyte	Standard IDs	L	M1	M2	M3	H
AR1660	4566-4570	0.1875	0.375	0.75	1.5	3.0
AR1221	4571-4575	0.25	0.50	1.0	2.0	4.0
AR1232	4576-4585	0.1875	0.375	0.75	1.5	3.0
AR1242	4581-4585	0.125	0.25	0.50	1.0	2.0
AR1248	4605-4609	0.125	0.25	0.50	1.0	2.0
AR1254	4610-4614	0.125	0.25	0.50	1.0	2.0
TCMX		0.005	0.01	0.02	0.08	0.16
DCBP		0.01	0.02	0.04	0.16	0.32

10.1.1.1 For samples analyzed in compliance with the DoD-QSM, the average calibration factor from a five-point initial calibration curve must be used to quantitate Aroclors 1221, 1232, 1242, 1248, and 1254.

10.1.1.2 If an Aroclor other than 1016 or 1260 is identified during the analysis of sample for DoD-QSM compliance, a five-point initial calibration curve must be analyzed. The affected samples must be reanalyzed for the Aroclor(s) indicated by the single-point analysis.

10.1.2 A minimum of five sets of calibration factors is generated for the Aroclor 1016/1260 standards. Each set consists of the calibration factor for each of the five peaks chosen. The single standard of the remaining Aroclors will each produce a minimum of three calibration factors, one for each selected peak.

10.1.3 The initial calibration standard sequence is as follows:

Injection Number	Calibration Standard
1	**AR1660-1**
2	**AR1660-2**
3	**AR1660-3**
4	**AR1660-4**
5	**AR1660-5**
6	**AR1221-M3**
7	**AR1232-M3**
8	**AR1242-M3**
9	**AR1248-M3**
10	**AR1254-M3**

10.2 Initial Calibration Verification (ICV)

10.2.1 The initial calibration is verified using a standard prepared from a separate source than that used for the calibration curve. An acceptable recovery must be achieved before proceeding with analysis. **(See Section 9.3.)**

10.3 Continuing Calibration

10.3.1 The continuing calibration verification standards are used to evaluate retention time stability.

10.3.2 For continuing calibration verification, the mid level Aroclor 1660 from the initial calibration is injected. Sequencing should be as follows.

12 Hour Sequence Injection No.[1]	Whole Sequence Bottle No.[3]	Description
1	6	**AR1660-M3D**[2]
2-**11**	7-**16**	**10 Samples**
12-17	**17**	**AR1660-M3**
18-**22**	**18-27**	**10 Samples**
23	**28**	AR1660-M3

[1] The number in the first column represents the injection sequence at the start of a twelve-hour time period. The number in the second column represents the actual bottle number in the injection sequence that would have begun at the start of the initial calibration. This example is based upon 15 sample injections between calibration standards.

[2] The "D" indicates this standard to be the "daily" or first calibration verification standard run during a 24 hour period. The AR1660-3D is the mid-level, Aroclor standard and the absolute retention time for each chosen characteristic Aroclor component peak establishes the midpoint of the retention time window.

[3] The requirements of Method 8082 include a calibration standard after each 20 samples although one is recommended after every 10 samples. The method also indicates that the calibration verification standards be run each 12 hour shift. The method does not specify the requirement or the frequency for instrument blanks. These are important tools for assessing instrument performance. Assuming approximately 30 minutes per analytical run, the 12-hour shift begins with the injection of the AR1660-3D standard (Injection No. **1**, bottle **6**). The 12-hour sequence period would end after Injection No. **23**, bottle **28** in the total sequence. At the end of the next 12-hour sequence, the closing AR1660-3 standard would also represent the start of the next 24-hour period so that standard would be used to establish the midpoint of the retention time windows for that period. If other Aroclors are detected during the analytical sequence they may be injected to replace an AR1660 calibration verification standard.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Dual Column Analysis and Confirmation

11.1.1 The dual column/dual detector approach involves the use of two 30 m x 0.53 mm ID fused silica open-tubular columns of different polarities (CLPesticides and CLPesticides2). Each has different selectivities towards the target compounds. The columns are connected to a "Y-splitter" and dual ECD detectors.

11.2 GC operating conditions are as follows:

11.2.1 Column 1

- Type: CLPesticides
- Dimensions: 30 m x 0.53 mm ID
- Film Thickness: 0.50 μm

11.2.2 Column 2

- Type: CLPesticides2
- Dimensions: 30 m x 0.53 mm ID
- Film Thickness: 0.42 μm

11.2.3 Carrier gas

- Helium
- Flow rate: 6 mL/minute

11.2.4 Makeup gas

- Argon/methane
- Flow rate 20 mL/minute

11.2.5 Temperature program

- Hold at 150° C for 1.0 minute

- Ramp to 275° C at 5 degrees/minute
- Hold for 2.0 minutes

11.2.6 Injector temperature: 250° C

11.2.7 Detector temperature: 300° C

11.2.8 Injection volume: 2 or 4 μL (1 or 2 μL per column)

11.2.9 Solvent

- Hexane

11.2.10 Detector: Dual ECD's

11.2.11 Range: 10

11.3 Analytical Sequence

11.3.1 The analytical sequence is discussed in Section 10 above. All information is recorded on the run log for the instrument (Attachment 3)

11.4 Gas chromatographic analysis:

11.4.1 The GC is set up according to the operating conditions detailed above. A final temperature of 240 - 275^{o} C is necessary to elute decachlorobiphenyl.

11.4.2 The initial calibration is performed according to the discussion in Section 10.0. The initial calibration is verified once using a second source standard (ICV).

11.4.3 A continuing calibration standard is run during each 12 hour shift using a mid-level calibration standard. Calibration standards are also injected after every 10 samples (although it is required after every 20 samples) and at the end of the analytical sequence. Since an analytical sequence may continue as long as instrumental QC criteria are met, the end of one 12-hour sequence is considered to be the beginning of the next 12-hour sequence. The calibration factor must meet acceptance criteria when compared to the initial calibration curve. When this criteria is exceeded, corrective action is required as described in Section 7.0 and may include

re-injection of the calibration verification standard or analysis of a new initial calibration before more samples are analyzed.

11.4.4 Each sample analysis must be bracketed with an acceptable initial calibration, continuing calibration verification standards (each 12 hr shift), or calibration standards interspersed with the samples. If a "closing" standard, which is injected after a group of samples fails to meet criteria due to being more than 15% above the mean calibration factor from the initial calibration and the Aroclor was not detected in that group of samples, then the samples do not require reinjection. However, if the standard is more than 15% below the mean calibration factor from the initial calibration, then re-injection is required.

11.5 Retention Time Windows

11.5.1 Retention time windows are established for each chosen characteristic Aroclor component (peak). The width of the retention time windows is based on actual retention times of selected peaks in the standards that are assessed over a 72-hour period. A standard deviation **is** calculated for each component.

11.5.2 For each Aroclor component chosen (3-5 peaks)**,** the absolute retention time from each "daily" standard is used as the midpoint of the window. A ± three times standard deviation value is applied to the absolute retention time. Windows are defined as ± three times the standard deviation of the absolute retention times of each chosen Aroclor component. Analyst experience is critical in the interpretations of the chromatograms.

11.6 Identification and Quantitation

11.6.1 Compound identification is based on a second column confirmation, with the second column being of a dissimilar nature. Target analyte peaks must fall within the retention time windows on both columns.

11.6.2 Tentative identification of an analyte occurs when three or more of the chosen Aroclor component peaks from a sample extract fall within the retention time windows and the pattern matches that of the Aroclor standard.

11.6.**3** Quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding peaks in the calibration

standard at the same retention times. The external calibration method is used for quantitation.

11.6.**4** When samples contain more than one multi-component analyte, a more experienced analyst may be required to process or assess the data. Similar conditions exist when multicomponent analytes have been subjected to environmental degradation, resulting in weathered Aroclors that may have significant differences in peak patterns than the standards.

11.6.**5** Use the external calibration procedure to establish mean response factors for each chosen Aroclor component by dividing peak area in the standard by the mass injected. Peak areas from peaks within the chosen Aroclor component's retention time window are used to calculate the quantity of each target Aroclor while retention times are used to identify each component.

11.6.6 The analyte concentration reported is the higher of the two concentrations detected on the dual column analyses, unless overlapping peaks are interfering with the accurate quantitation of the analytes. If interferences are present, the analyte concentration is reported from the column without the interferences. If there is > 40% difference between the two columns, the data is flagged with a "P" and discussed in the narrative.

11.6.6.1 To meet the requirements of the DoD-QSM, the narrative must reference the "Q" flag for results with a > 40% difference between the columns.

11.6.7 The Aroclors are quantitated using responses of five major peaks (minimum of three) of the sample pattern compared to the calibration factors of the same peaks in standard. Those concentrations are averaged to arrive at the reported concentration.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ \mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Calibration factors are calculated by dividing the total peak area for each chosen Aroclor component in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.9 Concentration

12.9.1 Concentration of aqueous samples

$$\mu g / L = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Vo)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract (µl)
Df = dilution factor. If no dilution, Df = 1.0
$\overline{CF}$ = mean calibration factor from the initial calibration
Vo = volume of water sample extracted (ml)
Vi = volume of extract injected (µl)

12.9.2 Concentration of soil samples (dry weight basis)

$$\mu g / kg = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams
D (dry weight)= $\frac{100 - \%\ moisture}{100}$

Ax, Vt, Df, $\overline{CF}$, Vi have the same definitions as for water.

12.9.3 Concentration of soil samples (dry weight basis) by GC using linear regression analysis:

$$x = \frac{(y - b)}{a}$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.10 Calculating Dilutions

12.10.1 If the response of the majority of the chosen Aroclor components exceeds the on-column amount of the high level Aroclor 1660 standard, it must be diluted and re-analyzed. Comparing the on-column nanogram amount of the chosen Aroclor components with the nanogram amounts of the same chosen Aroclor components from the high level standard used in the initial calibration will allow you to determine an appropriate dilution.

12.10.2 If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places

pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 8082

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998), Method 1080

16.**3** QCSOP: Proper Documentation Procedures

16.**4** QCSOP: Numerical Data Reduction

16.**5** "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.**6** Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.**7** NELAC Standards, approved July 2003, plus revisions

16.**8** QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.**9** New York State Environmental Laboratory Approval Program, Certification Manual, April 2005, plus revisions.

16.**10** CompuChem Quality Manual, Revision **7**, Update 1, **12**/**13**/05, plus revisions

16.**11** Sample Control SOP 4.1, "Receiving Samples"

16.**12** Sample Control SOP 4.6, "Storing Samples"

16.**13** Sample Preparation Procedure –1049, "PCBs in Water Preparation Procedure"

16.**14** Sample Preparation Procedure –735 (-233→945), "Low Level Preparation for Analysis of PCBs only in Soil/Sediment/Sludge"

16.**15** Sample Preparation Procedure –236, "Soxhlet Extraction of S/S/S Samples by Method 3540C in SW846 & NYSASP"

16.**16** Sample Preparation Procedure –247, "Automated Soxhlet Extraction of Soil/Sediment/Sludge and Wipe Samples by SW-846 + NYSASP."

16.17 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Method **Reporting Limits**

17.2 Attachment 2 – Instrument Maintenance Log

17.3 Attachment 3 – Instrument Run Log

Attachment 1

Method 8082 Reporting Limits

Aroclor	µg/L	µg/Kg	Ohio VAP µg/L	Ohio VAP µg/Kg
1016	**0.93**	**31**	**1.0**	**33.3**
1260	**0.93**	**31**	**1.0**	**33.3**
1242	**0.625**	**21**	**1.0**	**33.3**
1232	**0.93**	**31**	**1.0**	**33.3**
1221	**1.25**	**42**	**2.0**	**33.3**
1248	**0.625**	**21**	**1.0**	**33.3**
1254	**0.625**	**21**	**1.0**	**33.3**

Attachment 2

CompuChem a division of Liberty Analytical Corp. Logbook 4 H(4) 1

GC Extractables Instrument Maintenance Log: 66/67

Date: _____ Time:_____

System: _____

Problem: _____

_____

_____

_____

_____

Action Taken: _____

_____

_____

_____

_____

_____

Parts used:

Part Number	Description	Quantity

File Name Indicating Instrument In Control: _____

Performed By: _____ Date: _____

Reviewed By: _____ Date: _____

2/11/00:mlj

Attachment 3

COMPUCHEM a division of Liberty Analytical Corp Instrument ID 80/81 Sequence ID: _____ DATE ___/___/___

GC EXTRACTABLES RUN LOG

COMPUCHEM LOGBOOK 4 TTT 26 Amt.Inj. 2 µL (1 µL per column)

Method : 8081A 8082 8151A CLP SOM01.1 Other_____

		FILE NAME		DATE	CompuChem #	CASE/SDG#	CHEMIST	COMMENTS(ETC.)/DISPOSITION
1				/ /				
2				/ /				
3				/ /				
4				/ /				
5				/ /				
6				/ /				
7				/ /				
8				/ /				
9				/ /				
10				/ /				
11				/ /				
12				/ /				
13				/ /				
14				/ /				
15				/ /				
16				/ /				
17				/ /				
18				/ /				
19				/ /				
20				/ /				
21				/ /				
22				/ /				
23				/ /				
24				/ /				

SUPERVISOR APPROVAL_____

DATE _____

Hexane Lot No_____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

1/26/06:jad

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-735 (-233->-945) SOP Section #: 2.2.5.2 Revision #: 7

SOP Title: Low Level Preparation for the Analysis of PCBs Only in Soil/Sediment/Sludge by SW-846 and NYSASP

Effective date: (QA fills in) 3/19/04

- Procedure prepared by: Linda Carter Date: 3/12/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane C. Ellmore Date: 3/12/04
- Reason for change: SC DHEC compliance
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, 12/96, Method 3350B; New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above) ORIGINAL

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 2/11/05

Annual Review—Signature: _____ Date: 5/3/06

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -735 (-233-->945) Low Level Preparation for the Analysis of PCBs Only in Soil/Sediment/Sludge by SW-846 and NYSASP

Table of Contents

Sample Preparation Procedure -735 (-233-->945) Low Level Preparation for the Analysis of PCBs Only in Soil/Sediment/Sludge by SW-846 and NYSASP

1.0 Scope and Application

The following sample preparation procedure is designed to prepare soil/sediment/sludge samples for Aroclor-only analysis by GC/ECD. The soil samples are extracted by sonication and the extracts subjected to an acid wash to remove interferences. The procedure is based on SW-846, Method 3550B and Method 3665A (sulfuric acid wash).

The method detection limits (MDLs) and reporting limits are shown in Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

A 30-g aliquot of sample is spiked with the surrogate solution and then mixed with sodium sulfate, and extracted with a 1:1 methylene chloride/distilled acetone solvent mixture by sonication. The extract is then filtered, dried, and concentrated by Kuderna Danish (K-D) evaporation flask. Mandatory sulfuric acid wash of the extract is then performed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.3 Reporting Units – µg/Kg

3.4 An SDG is defined by the following, whichever is more frequent:

- Each 20 field samples received within a case, or
- Each 7 calendar day period (14 calendar days if requested by the client) during which field samples in a case are received (period beginning with the receipt of the first sample in the SDG)

NOTE: The Army Corps of Engineers **(US ACE) and SC DHEC** do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **and 10% for SC DHEC**. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

4.0 Interferences

4.1 Method interferences might be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatograms (GC). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the site being sampled. The cleanup procedures must be used to remove such interferences in order to achieve the Contract Required Quantitation Limits (CRQL) or Practical Quantitation Limits (PQL). Extract cleanup procedures include a mandatory sulfuric acid wash (Method 3665A) and the optimal gel permeatim cleanup (Method 3640A).

5.0 Safety

5.1 Wear the proper personal safety equipment (gloves, lab coat, safety glasses) while performing this procedure.

Laboratory staff is encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Sonic cell disruptor--Heat Systems, Ultrasonics, Inc., Model W-990 (475-watt with pulsing capability, No. 207 3/4-inch tapped disrupter horn), or equivalent device with a minimum 375-watt output capability.

NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. Erosion of the tip is indicated by a rough surface.

6.2 Sonabox (or equivalent)--for use with disrupter to decrease noise level

6.3 250-ml centrifuge bottles--Corning 1250 bottle, Corning glass

6.4 K-D apparatus

6.4.1 Concentrator tube--10-ml, graduated (Kontes K-570040-1029, or equivalent)

6.4.2 Evaporative flask--500-ml (Kontes K-470001-0500, or equivalent)

6.4.3 Snyder column--three-ball macro (Kontes K-503000-0121, or equivalent)

6.5 Funnels and Filter Paper

6.5.1 Powder funnels--10-cm diameter (optional), for filtration/drying

6.5.2 Filter paper--No. 41 pre-prepped Whatman (or equivalent), 9-cm circles (optional)

6.6 Silicon carbide boiling chips--approximately 10 to 40 mesh. Heat the chips to 400°C for 30 min or solvent rinse before use.

6.7 Water bath--heated, with concentric ring cover, capable of temperature control (±5°C).

NOTE: Always use the water bath in a hood.

6.8 Top-loading balance--capable of weighing accurately to ± 0.01 g

6.9 Nitrogen evaporation device--equipped with a heated bath that can be maintained at 35°C to 40°C, N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent)

6.10 Vials and caps--2-ml for GC autosampler

6.11 Bottle or test tube--20-ml with Teflon-lined screw-cap for sulfur removal, if needed.

6.12 Glass vials--minimum of 20-ml, with screw-cap and Teflon or aluminum foil liner

6.13 Spatula--stainless steel

6.14 Pipet--Volumetric 1.0-ml or 2.0-ml (optional)

6.15 Syringe--1.0-ml or 2.0-ml (optional)

6.16 Vials--10-ml, with aluminum crimp-top and Teflon-faced silicone rubber seal

6.17 Tube--centrifuge, 20- to 15-ml with 19-mm ground glass joint

6.18 Centrifuge--table top (IEC model Centra-8)

6.19 Vortex mixer--Genie, Model 550-6, Scientific Industrial, Inc., Bohemia, NY, or equivalent.

6.20 Disposable Pasture glass pipets, 1-ml.

7.0 Reagents and Standards

Refer to the Standards Preparation Logbook 22 F for details on preparation of standards used in this procedure. All standards used in this procedure are prepared in pesticide grade acetone or methanol. Standards are stored separately from samples in the laboratory when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in

this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Sodium sulfate--granular, anhydrous reagent grade, heated at 400°C for 4 hr, cooled, and stored in a closed glass bottle. Mallinckrodt anhydrous granular, or equivalent.

CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

7.3 Solvents - pesticide quality or equivalent. Each lot of solvent is tested to demonstrate that it is free of interferences before use.

7.3.1 Methylene chloride - must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid.

7.3.2 Hexane

7.3.3 Acetone

7.4 Surrogate solution #426 - The surrogates, tetrachloro-m-xylene and decachlorobiphenyl are added to all, samples, MS/MSD, LCS and blanks.

7.4.1 This solution must be prepared every six months or sooner if comparison with quality control check samples indicates degradation or concentration of solution compounds.

7.4.2 The surrogate solution is stored at 2-4.4°C when not in use.

7.5 PCB matrix spiking solution #4615-- Prepare a spiking solution in acetone or methanol that contains the following Aroclor at the concentration specified:

PCB	µg/ml
1660	5.0

7.5.1 This solution must be prepared every six months or sooner if comparison with quality control check samples indicates degradation or concentration of solution compounds.

7.5.2 The matrix spiking solution is provided by the Organic Standards Chemist and stored at 2-4.4 °C.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples are collected in 8 oz. wide mouth jars with PTFE – lined caps. They are obtained from the custodian out of cold storage.

8.3 Samples are stored at 2-4.4°C. They are obtained from the Custodian out of cold storage. Samples should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

8.4 Samples must be extracted within 14 days of collection.

9.0 Quality Control

9.1 **Method Blank**

9.1.1 Prepare a method blank with each extraction batch of up to 20 samples. For SC DHEC, the method blank is performed at a frequency of 10%.

9.2 **Laboratory Control Sample**

9.2.1 Prepare a laboratory control sample (LCS; blank spike, BS) with each extraction batch of up to 20 samples. **For SC DHEC, the laboratory control sample is performed at a frequency of 10%.** For NYSASP, the LCS is called a matrix spike blank.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike (MS) and matrix spike duplicate (MSD) are prepared for every sample delivery group (SDG). For SC DHEC, the matrix spike/matrix spike duplicate are performed at a frequency of 5% for soil samples.

9.4 Duplicates

9.4.1 Duplicates, at a frequency of **5**%, are required when processing **soil** samples submitted to meet the regulatory requirements of **South** Carolina D**H**E**C. This requirement may be satisfied with the MS/MSD.**

10.0 Calibration & Standardization

10.1 Ensure the balance has been calibrated for the day before use. Refer to Quality Control SOP 13.16 "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

11.1 Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase. Samples must be at room temperature.

11.2 The technician performing the sample extraction completes the Extraction Worksheet (Attachment 2). The worksheet accompanies the samples for acid washing (SPP -945) and then to the GC laboratory. Also, a witness observes the addition of the surrogate and spike solutions and signs off on the Extraction Worksheet, verifying that the additions were done correctly. Include on the worksheet, the manufacturers and lot numbers of the reagents/solvents used.

11.3 Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.4 Extraction with Sonication

11.4.1 Weigh approximately 30 g of sample into a 250-ml centrifuge bottle (record weight to the nearest 0.1 g on the Extraction Worksheet, and add 60 g of anhydrous sodium sulfate (granular). Mix the sample thoroughly.

11.4.2 For a sample to be used for the MS and MSD analysis, weigh out two additional 30-g portions of sample, (record weight to nearest 0.1 g), add 60 g of sodium sulfate, and add 1.0 ml of the **PCB** matrix spike solution #4615 to the MS and MSD aliquot. To prepare a LCS, weigh an additional 30.0 g of sodium sulfate (furnaced), and add 60.0 g sodium sulfate. Add 1.0 ml of **PCB** matrix spike solution #4615 to the LCS.

11.4.3 Prepare a method blank with each batch of soil/sediment samples extracted. A method blank for soil/sediment samples consists of 90 g of sodium sulfate spiked with the 2.0 ml of the surrogate solution #426 and is carried through the entire analytical procedure.

11.4.4 Add 2.0 ml of surrogate solution #426 to all samples, the MS, MSD, LCS, and blanks by using a volumetric pipet or a syringe. Mix the

solution well. The sample and the added sodium sulfate should be a homogeneous, granular mixture at this point.

11.4.5 Immediately add 80-100 ml of 1:1 methylene chloride acetone to the sample.

11.4.6 Place the bottom surface of the sonicator probe about 1/2 inch below the surface of the solvent but above the sediment layer.

11.4.7 Sonicate for 3 minutes using a 3/4-inch horn at 100% power output with PULSE set on 1 second, ON, and PERCENT DUTY CYCLE knob set at 50%. Do NOT use a microtip.

11.4.8 Centrifuge each sample for 2 minutes at 2000 rpm. Decant and filter the extracts through pre-prepped Whatman No. 41 filter paper in a powder funnel into the K-D apparatus. An alternative is a Buchner funnel filtration with Whatman No. 41 filter paper.

11.4.9 Repeat the extraction two more times with additional 80-100 ml portions of the 1:1 methylene chloride:DI acetone. Before each extraction, thoroughly mix the solid residue, and make certain that the sodium sulfate is free flowing and not a consolidated mass. As needed, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication by using the same funnel described in paragraph 11.4.8. After the final sonication, pour the entire sample into the funnel and rinse the beaker and funnel with 20-30 mL methylene chloride.

11.4.9.1 If particulate matter is observed, filter the entire extracted sample again by placing a new prepped filter in the original funnel and pouring the extracted sample through the funnel. Rinse with methylene chloride.

11.5 Soil Extract Concentration

11.5.1 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro-Snyder column, while adding about 1 ml of methylene chloride to the top of the Snyder. Place the K-D apparatus on a hot water bath (80-85°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. Reduce

the volume of liquid to 3-5 ml. Remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.5.1.1 DO NOT ALLOW THE K-D EVAPORATOR TO GO DRY. If this should occur, the entire procedure must be begun again.

11.6 Solvent Exchange into Hexane

11.6.1 Momentarily remove the Snyder column. Add 60 ml of hexane and a new boiling chip, and reattach the Snyder column. Pre-wet the column by adding about 1 ml of hexane to the top. Concentrate the solvent extract as before. When the apparent volume of liquid reaches 3-5 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Alternatively, hexane may be added to the top of the Snyder column without allowing the K-D apparatus to cool.

11.6.1.1 DO NOT ALLOW THE K-D EVAPORATOR TO GO DRY. If this occurs, the entire procedure must be begun again.

11.6.2 Remove the K-D and Snyder column, then using the nitrogen evaporation (N-EVAP) technique or microsnyder concentration, concentrate the extract to a final volume of 5.0 mL.

11.6.3 Bottle up the extract that is now ready for sulfuric acid clean-up using Sample Preparation Procedure –945, "Sulfuric Acid Wash of PCB-only Hexane Extracts by SW846."

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 1) and precision and accuracy for single analyst (Attachment 3). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places

pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. 3rd Edition, Update III, 12/96, Method 3550B

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **approved May 2001**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **4** 12/1**0**/0**3**, plus revisions

16.13 Sample Control SOP 4.1, "Receiving Samples"

16.14 Sample Control SOP 4.6, "Storing Samples"

16.15 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.15 Instrument Procedure 194 (SOP Section 2.2.5.3) "Polychlorinated Biphenyls (PCBs) as Aroclors by GC/ECD – Capillary Column Technique – Method 8082 (SW 846 and NYSASP)."

16.16 Sample Preparation Procedure –945, "Sulfuric Acid Wash of PCB-only Hexane Extracts by SW846."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – MDL Study

17.2 Attachment 2 – Extraction Worksheet (-735)

17.3 Attachment 3 - Single Analyst Capability Study

Attachment 1

Study Date & Instrument: Various					GC PCB Method 3550B/8082 Soil (Sonication)									
					Using higher of two column MDLs									
				Based on 30 g sample weight, 5 mL extract volume, without GPC										
Aroclor	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Test Conc.	S.Dev.	MDL	Report Limit
(Study date)(Varian)	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
1016 (Prep 1/11/04) (Analysis 1/17/04)(86/87)	41.0	44.0	42.0	43.0	47.0	43.0	43.0	46.0	43.0	43.6	33.3	1.88	5.4	31
1260 (Prep 1/11/04) (Analysis 1/17/04)(86/87)	37.0	36.0	33.0	37.0	35.0	34.0	34.0	34.0	30.0	34.4	33.3	2.19	6.3	31
1242 (Prep 1/19/04) (Analysis 1/20/04)(86/87)	38.0	32.0	33.0	35.0	37.0	31.0	36.0	31.0	*	34.1	33.3	2.75	8.2	21
1232 (Prep 1/11/04) (Analysis 1/17/04)(86/87)	51.0	52.0	56.0	58.0	57.0	54.0	57.0	47.0	*	54.0	33.3	3.8	11.3	31
1221 (Prep 1/29/04) (Analysis 1/30/04)(86/87	70.0	73.0	69.0	66.0	71.0	72.0	66.0	71.0	73.0	70.1	66.7	2.67	7.7	42
1248 (Prep 1/9/04) (Analysis 1/18/04)(86/87)	38.0	43.0	45.0	48.0	43.0	39.0	41.0	48.0	37.0	42.4	33.3	4.07	11.8	21
1254 (Prep 1/9/04) (Analysis 1/18/04)(86/87)	37.0	34.0	32.0	36.0	35.0	34.0	38.0	35.0	36.0	35.2	33.3	1.79	5.18	21
* Data point eliminated using Dixon outlier test														

Note: Attachment is subject to change without notice.
Note: The estimated detection limit is 1/5 the reporting limit.

Attachment 2

CompuChem
EXTRACTION WORKSHEET
PCB ONLY IN S/S/S BY METHOD 3550A/3665 for 8082

ASSIGNED TO _____

EMPLOYEE ID # _____

-735 PCB's ONLY

DATE EXTRACTED/POSTED _____

	SAMPLE NUMBER	QC SAMPLE TYPE	SAMPLE WEIGHT (g)	FINAL VOLUME (ml)	COMMENTS
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					ACID WASH (3665A) 5 ML FINAL EXTRACT
13					VOLUME.
14					
15					
16					
17					
18					
19					
20					
21					
22					
23		BLK			
24		LCS			

	#	AMOUNT	LOT #	SURROGATE AND SPIKE ADDED BY:
SURROGATE	426	2.0 ml		_____ / _____
SPIKE	4615	1.0 ml		INITIALS DATE

FINAL VOLUME VERIFIED: _____

SUPERVISOR REVIEWED: _____

Analysts initials. Extract _____ **KD** _____ **N2**_____ **Bottle up** _____ **Acid Wash** _____ **Witness** _____/_____
Initials Date

Manufacaturers and lot numbers of reagents/solvents used _____

Note: Attachment is subject to change without notice.

Attachment 3

Analyst Capability Study

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: 2359
Study Date: 10/18/01
Method: 8081, Multi-component Pesticides & AR1660
Instrument/Column: Varian 35/CLPesticides II

Compound	TrueVal	Rep #1	Rep #2	Rep #3	Rep #4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	- 3SD	+3SD	SOP	RSD
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	%R	%R	ug/L	ug/L	ug/L	%R	%R	%RSD	%
Instrument: Varian 35 ECD															
Toxaphene	10	9.7	11	11	12	11	109	50-150	0.94	8.1	14	74	126	20	8.6
Technical Chlordane	2.0	2.0	2.3	2.1	2.2	2.2	108	50-150	0.13	1.8	2.5	82	118	20	6.0
AR1016	7.5	5.8	6.5	6.4	6.4	6.3	84	50-150	0.32	5.3	7.2	85	115	20	5.1
AR1260	7.5	5.2	5.8	6.0	6.1	5.8	77	50-150	0.40	4.6	7.0	79	121	20	7.0

Note: Attachment is subject to change without notice.

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SOP 1021 SOP Section #: 2.2.4.3 Revision #: 8

SOP Title: Extraction of TCLP Leachates for the Determination of Pest/PCBs by SW846 + NYSASP

Effective date: (QA fills in) 4/2/03

- Procedure prepared by: _____ Date: 3/19/03
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane E Elmore Date: 4/2/03
- Reason for change: LCS change
- This procedure meets the requirements of the following approved method references: SW846, 3rd Ed. Update III, 3510C; NYSASP 6/2000 plus rev.

Procedure approved by Quality Assurance Representative: _____ Date: _____
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: 1/14/04

Annual Review—Signature: _____ Date: 2/11/05

Annual Review—Signature: _____ Date: 5/3/06

Sample Preparation Procedure -1021: Extraction of TCLP Leachates for the Determination of Pesticides/PCBs by SW846 and NYSASP

Table of Contents

Sample Preparation Procedure -1021: Extraction of TCLP Leachates for the Determination of Pesticides/PCBs by SW846 and NYSASP

1.0 Scope and Application

This procedure is used to extract pesticides/PCBs from the aqueous leachate generated using Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)".

The method detection limits (MDL) and reporting limits are shown it Attachment 1.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Approximately 200 ml of a previously prepared TCLP leachate are diluted to 1000 ml (a 5:1 dilution), and extracted with methylene chloride using a separatory funnel (continuous liquid-liquid) technique. The methylene chloride extract is dried, exchanged to hexane, and adjusted to a final volume of 10.0 ml. Optional cleanup techniques may be needed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements.

3.3 Reporting Units – µg/L

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers **(US ACE) and South Carolina Department of Health and Environmental Control (SC DHEC)** do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% **for US ACE and 10% for SC DHEC**. If samples are batched together from different sites, project-specific QC must be processed.

4.0 Interferences

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware cause method interferences that lead to discrete artifacts and/or elevated baselines in gas chromatograms (GC). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector (ECD). These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoiding the use of plastics in the laboratory minimizes interference from phthalates. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the site being sampled.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Glassware

6.1.1 Separatory funnel, 2000-ml, with Teflon stopcock

6.1.2 Drying column: chromatographic column, approximately 400 mm x 19 mm ID, with a small plug of furnaced glass wool. A glass funnel with a small plug of glass wool may be substituted for the drying column. Furnaced anhydrous sodium sulfate is used in the drying column or glass funnel to dry the solvent extract.

6.1.3 Concentrator tube, Kuderna-Danish: 10-ml, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes used in the test.

6.1.4 Evaporative flask - Kuderna-Danish, 500-ml (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs, rubber band, or blue Keck clip.

6.1.5 Snyder column - three-ball macro (Kontes K-503000-0121 or equivalent).

6.1.6 1000 ml graduated cylinders

6.1.7 250 ml Erlenmeyer flask, glass

6.2 Pyrex glass wool. Anneal in a 500° C oven for 2-4 hours before use.

6.3 Silicon carbide boiling chips, approximately 10/40 mesh. Heat to 400° C for 30 minutes or Soxhlet extract with methylene chloride for 4 hours.

6.4 Water bath, heated with concentric ring cover, capable of temperature control to ± 2°cC. The bath should be used in a hood.

6.5 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40° C. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.

6.6 Wide range pH paper (0-14 pH range)

6.7 1.0 ml serological pipets

6.8 Pasteur pipets, glass, disposable

6.9 10-ml amber serum glass vial with crimp-top aluminum seal and Teflon-faced septa

7.0 Reagents & Standards

All standards are prepared by the Organic Standards chemist. Details for the preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.) Standards are stored at 4 ± 2°C when not in use.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Solvents, pesticide grade or equivalent

7.2.1 Hexane

7.2.2 Methylene chloride

7.3 Sodium sulfate, (ACS grade) granular, anhydrous.

7.3.1 Dry in 400° C oven for 4 hours in a shallow tray.

7.4 Sodium hydroxide solution (10N), ACS grade.

7.4.1 Dissolve 40 g of NaOH pellets in DI water and dilute to 100 ml.

7.5 Sulfuric acid solution, **concentrated**

7.6 Pesticide/PCB surrogate standard solution #426. The surrogate compounds are tetrachloro-m-zylene (TCX) and decachlorobiphenyl (DCB) at a concentration of 0.2 µg/ml methanol.

7.7 TCLP PEST/PCB spiking standard solution. The spiking solutions in methanol contain the following pesticides in the concentrations specified:

Compound	Concentration as µg/**m**L
Lindane	0.3
Heptachlor	0.3
Heptachlor epoxide	0.3
Toxaphene	10**.0**

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All TCLP leachates must be extracted within 7 days of leachate generation.

8.3 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

9.0 Quality Control

9.1 Method blank

9.1.1 A method blank must be prepared and analyzed with every batch of up to 20 samples extracted. **For SC DHEC analyze the blank every 10 samples.** The method blank is prepared using DI water.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS; blank spike, BS; matrix spike blank for NYSASP) must be prepared and analyzed with every batch of up to 20 samples extracted. **For SC DHEC analyze the LCS every 10 samples.** The LCS is prepared using **extraction fluid diluted with** DI water.

9.3 Matrix Spike and Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) must be performed for every SDG.

9.4 Duplicates

9.4.1 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of **SC DHEC**. The MS/MSD **count towards** this requirement.

10.0 Calibration & Standardization

NA

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The sample preparation technician must complete the extraction worksheet (Attachment 2). Any unused portions must be Z-d out. The laboratory supervisor reviews the completed worksheet for accuracy and completeness and then signs it. The worksheet accompanies the sample to the analytical laboratory. Include on the worksheet the manufacturer and lot number of the reagent/solvents used.

11.1 Prep (rinse) all glassware with methylene chloride before use, including the drying column containing sodium sulfate. Do not use any glassware that appears to be dirty or cracked. Return dirty glassware to the glassware prep area for washing, and place cracked glassware in a box to be sent for repair.

11.2 Thoroughly mix the sample before aliquotting. Using a one-liter graduated cylinder, measure 200 ml of the TCLP leachate. Record the volume on the extraction worksheet. Dilute to 1000 ml with DI water (5:1 dilution), and transfer to a 2 liter separatory funnel. Pipet 1.0 ml #426 surrogate standard spiking solution into the separatory funnel and mix well. Check the pH of the sample using wide range pH paper and adjust to a pH between 5 and 9 with **10N** sodium hydroxide solution or **concentrated** sulfuric acid.

11.3 For the MS/MSD follow step 11.2 using two additional aliquots from one original unspiked sample. Add 1.0 ml of TCLP PEST/PCB spike solution and 1.0 ml of #426 standard to the MS/MSD.

11.3.1 Alternately, 100 ml of sample may be diluted to 500 ml with DI water with 0.5 ml surrogate and/or spike standard added. The final volume is also reduced by half.

11.4 Prepare a method blank using 1000 ml extracted DI water and transferring to a 2-liter separatory funnel. Add 1.0 ml #426 standard.

11.5 Prepare a LCS by measuring **200 ml extraction fluid diluted to** 1000 ml **with** DI water and transferring to a 2 liter separatory funnel. Add 1.0 ml TCLP PEST/PCB solution and 1.0 ml #426 standard.

Alternatively, 100 ml of the leachate fluid may be diluted to 500 ml and surrogate, spike and final volume reduced by half.

11.6 To prepare the leachate blank, use 200 ml of the TCLP leachate blank and dilute to 1000 ml with water. Add 1.0 ml of surrogate solution #426.

Alternatively, 100 ml of the leachate blank may be diluted to 500 ml and surrogate and final volume reduced by half.

11.7 Add 60 ml of methylene chloride to each separatory funnel and extract the samples by shaking the funnel for two minutes, with periodic venting under the hood to release excess pressure. Allow the organic layer to separate from the water phase for at least 10 minutes. If the emulsion interface between layers is more than one third of the volume of the solvent layer, the technician must employ mechanical techniques to complete the phase separation. The optimum technique depends on the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means. Drain the methylene chloride into a 250-ml Erlenmeyer flask.

11.8 Add a second 60 ml volume of methylene chloride to the separatory funnel and repeat the extraction procedure, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.9 Assemble a 500-ml Kuderna-Danish (K-D) evaporative flask, a 10 ml concentrator tube, and a three-ball macro Snyder column. Prep, i.e. rinse, all glassware with methylene chloride.

11.10 Pour the combined extract through a drying column containing about 10 cm of furnaced sodium sulfate, and collect the extract in the K-D apparatus. Rinse the Erlenmeyer flask and column with 20-30 ml of methylene chloride to complete the quantitative transfer.

11.11 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro Snyder column. Pre-wet the Snyder column by adding about 1.0 ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80 to 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 4 ml, remove the K-D apparatus from the bath. Allow it to drain and cool.

11.12 After the sample has cooled, remove the Snyder column. Add 50 ml hexane. Mix the solvent in the K-D apparatus by carefully tilting it and allowing the solvent to flow out of the attached concentrator tube. Gently shake the K-D apparatus to mix the solvents. Repeat the mixing step several times. Pre-wet the top of the Snyder column by adding about 1 ml of hexane. Replace the K-D apparatus on the hot water bath. When the apparent volume of liquid reaches 4 ml, remove the K-D apparatus. Allow it to drain and cool for at least ten minutes.

Caution: Never allow the extract to go dry during any concentration step.

11.13 Concentration technique

11.13.1 Remove the K-D and Snyder column, then place the concentrator tube in the N-EVAP and evaporate the solvent volume to 10.0 ml using a gentle stream of clean, dry nitrogen. Alternatively, microsnyder concentration may be used.

11.13.2 Rinse the internal wall of the tube several times with hexane as the extract concentrates.

11.14 Quantitatively transfer the contents of the concentrator tube to a 10 ml clear vial. Label the amber vial with a green label containing the following information:

CompuChem number
-1021
Date extracted

Complete all paperwork, verifying the final volumes, and obtain supervisor review of paperwork and extracts. Deliver the extracts and the associated paperwork to the designated area within the GC instrumentation laboratory.

11.16 The extract is now ready for analysis following Instrument Procedure 192, "Organochlorine Pesticides by GC/ECD-Capillary Column Technique, Method 8081A"

11.17 Florisil cleanup following Sample Preparation Procedure –938, "Automated Florisil Cartridge Cleanup for Pesticide/PCB Analysis by CLP, SW846, and NYSASP" may be performed if indicated by the GC laboratory after initial analysis.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 1) and precision and accuracy for single analyst (Attachment 3). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, 12/96,Method 3510C.

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 New York State Analytical Services Protocol (NYSASP), 6/2000, plus revisions.

16.4 QCSOP: Proper Documentation Procedures

16.5 QCSOP: Numerical Data Reduction

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, June 2000, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.11 CompuChem Quality Manual, Revision **4**, 12/1**0**/0**3**, plus revisions

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP)".

16.15 Instrument Procedure 192, "Organochlorine Pesticides by GC/ECD-Capillary Column Technique, Method 8081A"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Method detection limits (MDLs) and reporting limits

17.2 Attachment 2 – Sample Preparation –1021 Worksheet

17.3 Attachment 3 – Single Analyst Capability Study

Attachment 1

Study Date & Instrument: Various				GC Pesticide Method 608/8081A Aqueous										
				Using higher of two column MDLs										
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Amt.	S.Dev.	MDL	Report
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	Limit
Mix A(1/17/02)(43/46)														ug/L
alpha-BHC	0.0540	0.0580	0.0530	0.0550	0.0500	0.0540	0.0490	0.0560	0.0360	0.0517	0.0500	0.00650	0.019	0.10
gamma-BHC(Lindane)	0.0530	0.0540	0.0530	0.0550	0.0520	0.0570	0.0520	0.0600	0.0470	0.0537	0.0500	0.00361	0.010	0.050
Heptachlor	NA	0.0630	0.0710	0.0600	0.0570	0.0770	0.0520	0.0660	0.0500	0.0620	0.0500	0.00923	0.028	0.10
Endosulfan I	0.0230	0.0220	0.0210	0.0250	0.0210	0.0220	0.0200	0.0240	0.0200	0.0220	0.0500	0.00173	0.0050	0.10
Dieldrin	0.110	0.106	0.105	0.115	0.104	0.112	0.102	0.122	0.0980	0.108	0.1000	0.00733	0.021	0.10
Endrin	0.109	0.105	0.105	0.114	0.103	0.111	0.103	0.122	0.0970	0.108	0.1000	0.00733	0.021	0.20
4.4'-DDD	0.108	0.101	0.0980	0.108	0.0950	0.117	0.108	0.129	0.101	0.107	0.1000	0.0105	0.030	0.20
4,4-DDT	0.125	0.109	0.111	0.122	0.115	0.126	0.120	0.138	0.113	0.120	0.100	0.00912	0.026	0.30
Methoxychlor	0.545	0.508	0.517	0.580	0.500	0.550	0.520	0.601	0.491	0.535	0.500	0.0373	0.11	0.50
Mix B(1/17/02)(43/46)														
Aldrin	0.0550	0.0550	0.0510	0.0510	*	0.0530	0.0530	0.0540	0.0550	0.0534	0.050	0.00169	0.0051	0.050
Isodrin	0.500	0.503	0.467	0.461	*	0.467	0.486	0.491	0.512	0.486	0.50	0.0190	0.057	0.20
beta-BHC	0.0510	0.0520	0.0470	0.0330	*	0.0480	0.0490	0.0490	0.0510	0.0475	0.050	0.00609	0.018	0.10
d-BHC	0.0570	0.0580	0.0530	0.0340	0.0220	0.0540	0.0550	0.0540	0.0560	0.0492	0.050	0.0125	0.036	0.15
Heptachlor epoxide	0.0720	0.0620	0.0540	0.0510	*	0.0520	0.0550	0.0520	0.0590	0.0571	0.050	0.00710	0.021	0.10
g-chlordane	0.0580	0.0600	0.0540	0.0520	*	0.0550	0.0570	0.0560	0.0580	0.0563	0.050	0.00255	0.0076	0.050
alpha-chlordane	0.0550	0.0570	0.0520	0.0510	*	0.0530	0.0530	0.0550	0.0560	0.0540	0.050	0.00207	0.0062	0.10
4,4'-DDE	0.107	0.113	0.103	0.099	*	0.104	0.0980	0.0930	0.103	0.103	0.10	0.00605	0.018	0.10
Endosulfan II	0.0480	0.0490	0.0440	0.0450	*	0.0450	0.0420	0.0430	0.0490	0.0456	0.10	0.00272	0.0082	0.20
Endrin aldehyde	0.112	0.119	0.104	0.108	*	0.106	0.112	0.110	0.166	0.117	0.10	0.0203	0.061	0.20
Endosulfan sulfate	0.124	0.133	0.116	0.118	*	0.116	0.115	0.114	0.121	0.120	0.10	0.00635	0.019	0.20
Endrin ketone	0.122	0.129	0.112	0.107	*	0.115	0.117	0.123	0.123	0.119	0.10	0.00709	0.021	0.50
Tech.chlordane(3/14/02)(80/81)	0.620	0.570	0.550	1.00	0.650	NA	0.590	0.580	NA	0.651	0.50	0.157	0.494	5.0
Toxaphene(2/10/02)(43/46)	1.90	2.20	1.90	2.20	2.20	2.20	1.90	2.40	1.60	2.06	5.0	0.246	0.71	10.0

Attachment 1 (continued)

Study Date & Instrument: Various				GC PCB Method 608/8082 Aqueous										
				Using higher of two column MDLs										
Aroclor	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Rep#9	Mean	Amt.	S.Dev.	MDL	Report
(Study date)(Varian)	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	Limit
														ug/L
1016(1/29/02)(70)	1.80	2.00	1.70	2.20	1.90	2.20	2.00	2.00	2.00	1.98	2.0	0.164	0.48	1.5
1260(1/29/02)(70)	2.20	2.10	2.00	2.00	2.10	2.30	2.30	2.20	2.20	2.16	2.0	0.113	0.33	1.5
1242(1/30/02)(72)	0.970	0.910	0.890	0.810	1.00	0.880	1.00	0.810	1.30	0.952	1.0	0.149	0.43	1.5
1232(2/4/02)(70)	1.30	1.10	1.40	1.00	1.20	1.30	1.20	1.30	1.20	1.22	1.0	0.120	0.35	1.5
1221(1/28/02)(70)	2.20	1.90	1.40	2.30	1.60	2.20	1.50	2.00	2.00	1.90	2.0	0.328	0.95	3.0
1248(1/29/02)(70)	0.950	0.920	1.10	1.10	0.860	0.920	0.780	0.950	0.990	0.952	1.0	0.103	0.30	1.5
1254(2/11/02)(70)	1.40	1.50	1.20	1.50	1.50	1.60	1.30	1.60	1.40	1.44	1.0	0.133	0.39	1.5

Attachment 2

ASSIGNED TO: _____

EMP ID NUMBER: _____

COMPUCHEM
EXTRACTION WORKSHEET
Pesticide in Water; BY SW-846 Method 3510C
TCLP Waste Characterization - 8081A

DATE EXTRACTED: _____

-1021

	SAMPLE NUMBER	QC SAMPLE TYPE	SAMPLE VOLUME (ml)	FINAL VOLUME (ml)	COMMENTS
1					
2					
3					
4					Use 100 mL of TCLP leachate and dilute to 500 mL with extracted
5					water for all samples. Add 0.5 mL TCLP Pesticides spike
6					to SS's and BS. Add 0.5 mL of #426 Surr. To all samples.
7					Final volume = 5.0 mL.
8					_____
9					Florisil (3620B) performed Y / N
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23		TCLPBLK			
24		TCLPBLK			
25		BLK			
26		LCS			

		EXT. TPH	SURROGATE & SPIKE ADDED BY
	No.	426	_____ _____
SURROGATE	Amt.	0.5 ml	INITIALS / DATE
TCLP	No.		
PEST SPIKE	Amt.	0.5 ml	Witness _____/_____
			Initials Date

FINAL VOLUME VERIFIED _____

SUPERVISOR REVIEWED _____

Analysts Initials. Extracted_____ **KD** _____ **N2** _____ **Bottle up**_____

Manufacturer and lot number of reagents/solvents used _____

Attachment 3

Analyst Capability Study

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: 2359
Study Date: 10/17/01
Method: 8081, Pesticides
Instrument/Column: Varian 34/CL Pesticides I

Compound	TrueVal	Rep #1	Rep #2	Rep #3	Rep #4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	- 3SD	+3SD	SOP	RSD
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	%R	%R	ug/L	ug/L	ug/L	%R	%R	%RSD	%
Instrument: Varian 34															
ECD															
g-BHC (Lindane)	0.10	0.10	0.088	0.090	0.084	0.09	89	50-150	0.00	0.08	0.10	85	115	20	5.1
Heptachlor	0.10	0.10	0.10	0.10	0.094	0.099	99	50-150	0.00	0.09	0.11	91	109	20	3.0
Aldrin	0.10	0.095	0.088	0.090	0.083	0.089	89	50-150	0.00	0.07	0.10	83	117	20	5.6
Isodrin	0.10	0.094	0.089	0.089	0.083	0.089	89	50-150	0.00	0.08	0.10	85	115	20	5.1
g-Chlordane	0.10	0.094	0.089	0.090	0.083	0.089	89	50-150	0.00	0.08	0.10	85	115	20	5.1
Endosulfan I	0.20	0.19	0.18	0.18	0.16	0.18	89	50-150	0.01	0.14	0.22	79	121	20	7.1
Dieldrin	0.20	0.19	0.18	0.18	0.17	0.18	90	50-150	0.01	0.16	0.20	86	114	20	4.5
Endosulfan II	0.40	0.43	0.40	0.40	0.36	0.40	99	50-150	0.03	0.31	0.48	78	122	20	7.2
4,4'-DDT	0.60	0.66	0.60	0.61	0.55	0.61	101	50-150	0.05	0.47	0.74	78	122	20	7.5
Methoxychlor	1.0	1.1	1.0	1.1	0.96	1.0	104	50-150	0.07	0.83	1.3	79	121	20	6.8
a-BHC	0.10	0.11	0.088	0.093	0.096	0.097	97	50-150	0.01	0.07	0.13	71	129	20	9.7
b-BHC	0.20	0.21	0.17	0.19	0.19	0.19	95	50-150	0.02	0.14	0.24	74	126	20	8.6
d-BHC	0.10	0.12	0.092	0.098	0.098	0.10	102	50-150	0.01	0.07	0.14	64	136	20	12.1
Heptachlor epoxide	0.10	0.11	0.088	0.092	0.096	0.097	97	50-150	0.01	0.07	0.13	70	130	20	9.9
a-Chlordane	0.20	0.22	0.18	0.18	0.19	0.19	96	50-150	0.02	0.14	0.25	70	130	20	9.8
4.4'-DDE	0.20	0.24	0.19	0.20	0.20	0.21	104	50-150	0.02	0.14	0.27	68	132	20	10.7
Endrin	0.40	0.46	0.36	0.38	0.39	0.40	99	50-150	0.04	0.27	0.53	67	133	20	10.9
4,4'-DDD	0.40	0.50	0.39	0.41	0.42	0.43	108	50-150	0.05	0.29	0.57	66	134	20	11.2
Endrin aldehyde	0.40	0.44	0.35	0.36	0.38	0.38	96	50-150	0.04	0.26	0.50	68	132	20	10.5
Endosulfan sulfate	0.40	0.44	0.35	0.37	0.38	0.39	96	50-150	0.04	0.27	0.50	70	130	20	10.1
Endrin ketone	1.0	1.10	0.90	0.94	0.97	0.98	98	50-150	0.09	0.72	1.2	73	127	20	8.9

Analyst Capability Study

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: 2359
Study Date: 10/18/01
Method: 8081, Multi-component Pesticides & AR1660
Instrument/Column: Varian 35/CLPesticides II

Compound	TrueVal	Rep #1	Rep #2	Rep #3	Rep #4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	- 3SD	+3SD	SOP	RSD
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	%R	%R	ug/L	ug/L	ug/L	%R	%R	%RSD	%
Instrument: Varian 35															
ECD															
Toxaphene	10	9.7	11	11	12	11	109	50-150	0.94	8.1	14	74	126	20	8.6
Technical Chlordane	2.0	2.0	2.3	2.1	2.2	2.2	108	50-150	0.13	1.8	2.5	82	118	20	6.0
AR1016	7.5	5.8	6.5	6.4	6.4	6.3	84	50-150	0.32	5.3	7.2	85	115	20	5.1
AR1260	7.5	5.2	5.8	6.0	6.1	5.8	77	50-150	0.40	4.6	7.0	79	121	20	7.0

CompuChem
a division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure X outdated procedure (archive) _____

- Procedure Code: IP 481 B SOP Section #: 1.3.2.4 Revision #: 10

SOP Title: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment Samples by SW-846 and NYSASP

Effective date: (QA fills in) 2/8/05

- Procedure prepared by: Valgena Respass Date: 2/8/05
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 2-9-05
- Reason for change: added formulas to calculation section
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, Method 8260B and Method 5035; NY State Analytical Services Protocol (NYSASP), June 2000, plus revisions

JA 11/2/10

Procedure approved by Quality Assurance Representative: **(Not needed if signed above)** Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] Date: 5.15.06

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 and NYSASP

Table of Contents

Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 and NYSASP

1.0 Scope and Application

This is a general purpose procedure for the identification and simultaneous measurement of purgeable volatile organic compounds in a variety of solid matrices following Method 8260B and incorporating Method 5035. The method is applicable to a wide range of organic compounds. Target compounds that may be analyzed by this method are listed in Table 1, Attachment 1, along with their associated internal standards and quantitation ions. Note, however, that many of these compounds are not routinely analyzed.

Method detection limits (MDL) and reporting limits are shown in Attachment 3. The reporting limit is the low level calibration standard concentration.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Low concentration level volatiles in soil/sediment/sludge samples are analyzed using a closed system purge and trap technique (Method 5035). Field samples are collected in vials containing a preservative solution (sodium bisulfate), immediately sealed and shipped to the laboratory at 4°C ± 2°C. Alternatively, an EnCore sampling device (or equivalent) is used to obtain a 5g or 25g sample that does not contain headspace. When the EnCore device is used, it is sent refrigerated to the laboratory and, within 48 hours of sampling, a weighed aliquot must be transferred to a vial containing a sodium bisulfate solution.

A special autosampler is used for the analysis. This device allows the sample vial to remain closed while reagent water and a solution containing internal standards and surrogates are injected by a needle through the septum. The device allows the mixture in the sample vial to be stirred, using a magnetic stir bar. The needle used to pierce the septum to deliver the water, internal standards, and surrogates is then the source of an inert gas which is introduced at the top of the sample vial. The same needle has entrance holes located above the sample/water level to collect and transfer the headspace onto a

sorbent column, where the purgeables are trapped. The autosampler also provides for heating the sample vial to 40°C while the contents are being stirred and the sample constituents purged and trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatograph (GC) wide-bore capillary column. The GC is temperature-programmed to separate the purgeables that are then detected with a mass spectrometer (MS).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics and the Contract Required Detection Limit (CRDL) for inorganics.

3.3 Reporting Units – ug/kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers (USACE) and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are

prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

4.0 Interferences

4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, it may be necessary to reduce solvent contamination in the laboratory, purge the methanol used to prepare standard solutions, purge the reagent water with helium or nitrogen, change the purge gas source, or regenerate the molecular sieve purge gas filter.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples are stored in GC/MS VOA laboratory refrigerator #5; separate from laboratory standards, and they must be analyzed in a room in which the atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. Because methylene chloride will permeate PTFE tubing, all GC carrier gas lines and purge gas plumbing are to be constructed from stainless steel or copper tubing.

4.3 Contamination by carryover can occur whenever a sample is analyzed after a sample that contains high levels of organic compounds. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank or the next sample must be closely monitored to check for cross-contamination. For samples containing large amounts of water soluble materials, high boiling compounds, or high purgeable levels, it may be necessary to clean the purge and trap apparatus by purging a 10-20% methanol solution, followed by baking the purge and trap apparatus and the analysis of an instrument blank to confirm that the system is free from contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 Instrument Problems/Preventative Maintenance

4.4.1 If a low response is observed for the early eluting compounds such as the gases, replacement of the trap or septum may be necessary. In addition, adjustments to the purge flow may be necessary to achieve a desired response for these compounds. If such adjustments do not help, it may be necessary to check the fittings on the purge and trap device and on the column for leaks. This is done with a helium leak detector and certain software utility programs.

4.4.2 Column maintenance or replacement may be necessary if peak tailing or broad chromatographic peaks are observed.

5.0 Safety

5.1 The toxicity and carcinogenicity of many chemicals used in this method have not been precisely determined: each chemical should be treated as a potential health hazard. Exposure to these chemicals should be minimized. Preparation of calibration standards, blanks, and samples is performed in a fume hood to minimize risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2-dichloroethane, 1,1,2,2-tetrachloroethane, 1,1,2- trichloroethane, chloroform, 1,2-dibromoethane, trichloroethene, and vinyl chloride.

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Syringes

6.1.1 5-mL Hamilton gastight syringe with Luerlock tip

6.1.2 10-mL Hamilton gastight syringe with Luerlock tip

6.1.3 25-mL Hamilton gastight syringe with Luerlock tip

6.1.4 10-µl Hamilton syringe

6.1.5 1-µl Hamilton syringe

6.2 Volumetric Flasks and Pipets

6.2.1 Assorted volumetric flasks ranging from 50-mL to 1000-mL

6.2.2 10-mL graduated pipet in 1/10-mL graduations

6.3 Vials

6.3.1 40 mL screw-top, PTFE-lined, septum-sealed vials, each containing a magnetic stirring bar.

6.4 Analytical Column

6.4.1 Supelco SPB-624 60-m, 0.32mm ID with 1.8 um film thickness

6.4.2 Restek RTX-VMS 20-m, 0.18mm ID with 1 um film thickness

6.5 Mass Spectrometer (MS)

6.5.1 The MS scans 35-300 amu at 0.7-sec scan time in the electron impact mode at 70 eV (nominal).

6.5.2 Hewlett Packard 5890 GC

6.5.3 Hewlett Packard 6890 GC

6.5.4 Hewlett Packard 5972 MSD

6.5.5 Finnigan INCOS 500 mass spectrometers

6.6 Interface (GC to MS)

6.6.1 Type:

6.6.1.1 Jet separator

6.6.1.2 Direct capillary interface

6.6.2 Temperature: 250°C

6.6.3 Alternate: column direct to MS

6.7 Data System

6.7.1 A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.7.2 The data processing software searches any GC/MS data file for ions of specified mass and plots abundance versus time or scan numbers. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software integrates the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, the software compares sample spectra against reference library spectra. The reference library used is the NIST Mass Spectral Library.

6.7.3 For data acquisition, The INCOS 500 systems use Prolab software on Pentium computers.

6.7.4 For data acquisition, the Hewlett Packard systems use ChemStation software on Pentium computers.

6.7.5 For data processing, the Hewlett Packard HP 9000 series 735 Unix Workstation employing HP ChemServer with Target3 and Envision software by Thru-Put Systems is used.

6.8 Gas Chromatograph

6.8.1 Varian 3400 conditions are listed below:

- Carrier Gas Helium
- GC mode Capillary

- Injection Port Temp 225°C
- Interface Temperature 225°
- Initial Temperature 0°C
- Final Temperature 185°C
- Column Flow Rate 25 ml/minute
  (10 ml/minute column flow +15 ml/minute makeup gas

6.8.2 The listed column flow rate is approximate. Flow rate is adjusted to optimize linear velocity of an unretained compound (butane) through the column. Optimum linear velocity for the column is 30 - 45 cm/second.

6.8.3 GC temperature program

6.8.3.1 This GC program is provided as an example; parameters may vary depending on the equipment.

- 0°C - 0°C for 2 minutes
- 0°C - 105°C @ 7°C/minute
- 105°C-185°C @ 26.7°C/minute
- 185°C-185°C
- Hold until all compounds elute.

6.9 Data Storage

6.9.1 Magnetic tape storage device: The magnetic tape storage device is capable of recording data and is suitable for long-term, off-line storage.

6.10 Purge and Trap Autosampler System

6.10.1 Tekmar LSC

6.10.1.1 Tekmar LSC 3000 with glass frit bottom liquid sample purging vessel and Luerlock valve.

6.10.1.2 The absorption trap must be at least 25 cm long and have an internal diameter of at least 0.105 inches (0.2667 cm).

6.10.2 Archon ALS

6.10.2.1 The Archon Model 5100, 4552 Purge and trap autosampler interfaces directly to a Tekmar 3000 Purge and Trap Concentrator.

6.10.2.2 The autosampler is designed for soil samples and utilizes 40 ml VOA vials with low bleed Teflon septa.

6.10.2.3 The Archon ALS has the capacity of up to 51 vials.

6.10.3 Trap Packing

6.10.3.1 Supelco "K" Trap

6.10.3.2 Carbopak B

6.10.3.3 Carboxen 1000 & 1001

7.0 Reagents and Standards

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methanol (B&J Scientific, purge and trap grade)

7.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent

7.4 Tuning Standard

7.4.1 Bromofluorobenzene - Standard ID# 7008 at 25 µg/mL. Two µl, yielding 50 ng on column, are injected onto the column every 12 hours.

7.4.1.1 Prepare the standard by adding 50µl Restek VOA Tuning Mix 5000 µg/ml) to an amount of purge and trap grade methanol in a 10 ml volumetric flask and bring to volume.

7.4.1.2 Prepare this standard monthly.

7.5 Calibration Standards

7.5.1 For the initial calibration, the internal standard solution is added automatically by the Archon Purge and Trap Autosampler. For all subsequent analyses, both the internal standard and the surrogate solutions are added automatically by the Archon autosampler.

Table 2: Standard Preparation

Std. ID TCL4-High	005 µg/kg	010 µg/kg	020 µg/kg	050* µg/kg	100 µg/kg	200 µg/kg
8260 I.S.	5.0 µl	5.0 µl	5.0 µl	5.0 µl	5.0 µl	5.0 µl
8260 S.S.	1.0 µl	2.0 µl	5.0 µl	10.0 µl	15.0 µl	20.0 µl
TCL4-1&2	1.0 µl	2.0 µl	4.0 µl	10.0 µl	20.0 µl	40.0 µl
TCL4-gases	1.0 µl	2.0 µl	4.0 µl	10.0 µl	20.0 µl	40.0 µl
TCL4-ketones	1.0 µl	2.0 µl	4.0 µl	10.0 µl	20.0 µl	40.0 µl
TCL4-AppIX	1.0 µl	2.0 µl	4.0 µl	10.0 µl	20.0 µl	40.0 µl

*Continuing Calibration level

7.5.1.1 To prepare the standards at the concentrations shown in the column headers of Table 2 above, add the µl amount of standard shown to a 100 ml volumetric flask containing nitrogen sparged DI water, then bring up to volume.

Alternatively, the standards may be prepared at the above concentrations by diluting the 200 µg/kg standard directly into the purge and trap impingers. Dilute as follows:

- For a 100 µg/kg standard, add 2.5 ml to 5 ml DI water;
- For a 50 µg/kg standard, add 1.25 ml to 5 ml DI water;
- For a 20 µg/kg standard, add 0.50 ml to 5 ml DI water;
- For a 10 µg/kg standard, add 0.25 ml to 5 ml DI water;
- For a 5 µg/kg standard, add 0.125 ml to 5 ml DI water.

7.5.1.2 The concentration of the compounds in the TCL4-1&2 High standard is 500 µg/ml. See Attachment 5 for the composition of this standard.

7.5.1.2.1 Prepare the standard by adding 1.25ml Restek 502.2 VOA 2000 MegaMix (2000 µg/ml) to an amount of purge and trap grade methanol in a 5 ml volumetric flask and bring to volume.

7.5.1.2.2 Prepare this standard every three months.

7.5.1.3 The concentration of the compounds in the TCL4-gases High standard is 500 µg/ml. See Attachment 5 for the composition of this standard.

7.5.1.3.1 Prepare each standard by adding 1.25ml Restek 502.2 Calibration Mix #1 (2000 µg/ml) to an amount of purge and trap grade methanol in a 5 ml volumetric flask and bring to volume.

7.5.1.3.2 The method states that this standard usually needs to be replaced weekly, unless the standard manufacturer recommends otherwise, or unless the acceptability of the standard can be documented. This standard has generally proven to be more stable in the laboratory. The gas standard can be used for longer than a week if the gases in the continuing calibration (CCV) standard meet the CCV requirements when compared to the initial calibration standards that contain a gas standard that has been prepared within a one-week holding time. Prepare this standard monthly, or more frequently as need dictates, or when degradation is evident rendering the standard unacceptable.

7.5.1.4 The concentration of the compounds in the TCL4-ketones standard is 500 ug/ml, 1250 ug/ml, or 5000 ug/ml. See Attachment 5 for the composition of this standard.

7.5.1.4.1 Prepare the standard by transferring AccuStandard custom VOA Mix #2 to a mininert vial.

7.5.1.4.2 Replenish this standard as needed and replace every three months.

7.5.1.5 The concentration of the compounds in the TCL4-AppIX standard is 500 ug/ml, 5000 ug/ml, or 25000 ug/ml. See Attachment 5 for the composition of this standard.

7.5.1.5.1 Prepare the standard by transferring AccuStandard custom VOA Mix #1 to a mininert vial.

7.5.1.5.2 Replenish this standard as needed and replace every three months.

7.6 Initial Calibration Verification

7.6.1 The initial calibration curve must be verified using a standard from an independent source. The laboratory purchases the initial calibration verification (ICV) standard from a different vendor than the one used for the calibration standards. This is prepared in the same manner as described above.

7.6.2 The ICV contains the full list of target analytes at the same concentration as the continuing calibration verification (CCV) standard.

7.7 Internal Standards

Compounds in 8260 I.S. (Internal standard) at a concentration of 50 µg/ml (250 ug/ml for the Archon)

- fluorobenzene
- D5-chlorobenzene
- D4-1,4-dichlorobenzene

7.7.1 Prepare the surrogate spiking solution by adding 0.2 ml Restek 8260A ampulated surrogate mix (2500µg/ml) to an amount of purge and trap grade methanol in a 10 ml volumetric flask and bring to volume.

7.7.2 Prepare this standard every three months.

7.8 Surrogate Standard

Compounds in 8260 S.S. (Surrogate standard) at a concentration of 50 µg/ml (250 **µ**g/ml for the Archon)

- dibromofluoromethane
- D4-1,2-dichloroethane
- D8-toluene
- 4-bromofluorobenzene

7.8.1 Prepare the surrogate spiking solution by adding 0.2 ml Restek 8260A ampulated surrogate mix (2500µg/ml) to an amount of purge and trap grade methanol in a 10 ml volumetric flask and bring to volume.

7.8.2 Prepare this standard every three months.

7.9 Spike Standard

Compounds in 8260 Spiking Mixture (1001C) at a concentration of 25 µg/mL

- 1,1-dichloroethene
- trichloroethene
- benzene
- toluene
- chlorobenzene

Note: The spiking cocktail is project dependent. This spiking mixture can also be used as a Laboratory Control Sample (LCS) spike. Some projects may require full analyte spike, and in that case, the standard used for the full analyte spike LCS is the ICV. For some programs, the CCV may be used in the place of the LCS.

7.9.1 Prepare this standard by adding 100 µl Restek VOA Matrix Spike Mix to an amount of purge and trap grade methanol in a 10 ml volumetric flask and bring to volume.

7.9.2 Prepare this standard every six months.

7.10 Standard Storage

7.10.1 Store the stock standards in Teflon- sealed screw-cap bottles with zero headspace at -10°C to -20°C. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases should be monitored and fresh standards prepared if a 20% difference/drift is experienced. These standards need to be replaced after six months or as recommended by the

manufacturer, unless the acceptability of the standard can be documented. CEVE and styrene may have to be prepared more frequently.

7.10.2 Store secondary dilution standards in Teflon-sealed screw-cap bottles with minimal headspace at -10°C to -20°C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards. Prepared from stock solution, they are stored with minimal headspace and replaced after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented.

7.10.3 Working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device. If premixed certified solutions are used store according to manufacturer's documented holding time and storage temperature recommendations.

7.10.4 Purgeable standards are stored in GC/MS VOA Freezer #1, separate from other standards and samples.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOPs 4.1, “Receiving Samples” and 4.6, “Storing Samples.” Sample holding times are also listed.

8.1.1 Note that if 2-chloroethyl vinyl ether is a target compound of interest for the project, an unpreserved sample must be analyzed within 7 days of collection.

8.2 All samples must be analyzed within 14 days of collection.

NOTE: For NYSASP all samples must be analyzed within 10 days of collection.

8.3 Prior to analysis, all samples must be stored under refrigeration at 2-4.4^{o} C in the reach-in storage unit in the laboratory. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogate compounds are added to all samples, QC, and standards prior to analysis. Surrogates are used to assess the efficiency of the analytical system.

9.1.2 Surrogate compounds must meet recovery criteria as shown below (Table 3).

Surrogate Compound	Soil % Recovery Range
Dibromofluoromethane	33-150%
D4-1,2-dichloroethane	43-145%
D8-toluene	55-125%
4-bromofluorobenzene	46-150%

9.1.2.1 For the NYSASP, the system monitoring compound (surrogate) recovery criteria are those in the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics, shown in the following table (Table 4).

The same surrogates and recovery criteria are to be used for samples submitted to meet the regulatory requirements of the State of South Carolina.

Surrogate Compound	Soil % Recovery Range
D4-1,2-dichloroethane	70-121%
D8-toluene	84-138%
bromofluorobenzene	59-113%

9.1.3 Samples with surrogate recovery failures must be re-extracted to confirm a matrix effect. Surrogate recovery failures in method blanks and LCS reqire re-extraction of the entire batch. Similar surrogate failures in the MS/MSD and original sample confirm a matrix effect and do not require re-extraction.

9.2 Internal Standards

9.2.1 The integrated areas of the quantitation ions of the internal standards are monitored in continuing calibration verification checks, samples, and QC

for a change in retention time and response or sensitivity. These should remain reasonably constant over time.

Internal standard retention time and area responses must be assessed in each continuing calibration verification standard by comparison to the corresponding internal standard in the most recent initial calibration mid-point standard. Internal standard responses in samples and QC are compared to the most recent continuing calibration verification.

9.2.2 The area responses of the internal standards must be within 50-200% difference of the area responses compared to.

9.2.3 The retention time for the internal standards must be less than 30 seconds.

9.2.4 If any of these criteria cannot be met, the analytical system must be checked for malfunctions and corrections made. Re-analysis of any affected sample is required.

9.3 Method/Instrument Blanks

9.3.1 Before any samples are analyzed, it must be demonstrated that a laboratory reagent blank is free from contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbents, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next analysis. In general, background from method analytes should be below the reporting limit.

9.3.2 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration verification acceptance criteria.

9.3.3 A method blank is analyzed with each batch of up to 20 samples processed as a group within a 12-hour tune. If more than 20 samples are analyzed in a tune batch, a second method blank is required. Method blanks must be analyzed immediately following a valid continuing calibration verification analysis. For SC DHEC a blank is required every 10 samples.

9.3.4 The concentration of the target compounds in the blank must be less than the reporting limit for each target compound. Except for SC DHEC the common lab solvents, methylene chloride and acetone, are allowed to be present at less than twice the reporting limit.

For NYSASP, the contamination criteria are those presented in the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics.

9.3.5 All samples processed within the same 12-hour tune associated with a method blank that does not meet the blank technical acceptance criteria must be reanalyzed.

9.3.6 Method interferences caused by contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms must be eliminated. The chromatographic system must be inspected for malfunctions, and corrections must be made as required before more samples are analyzed. An instrument blank is analyzed after a high concentration sample in order to eliminate carryover.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS, or blank spike, BS, or matrix spike blank for NYSASP) is prepared and analyzed with each tune batch of up to 20 samples. The LCS and matrix spike are spiked with the same target analytes. For SC DHEC the LCS is analyzed every 10 samples.

9.4.2 The solid LCS is prepared at 50 µg/kg with 50 µg/kg of surrogates.

9.4.3 The percent recovery criteria, developed from in-house statistical data, for an optional subset are shown below. Statistical control limits for the remainder of the analytes in the full LCS are listed in Attachment 2.

LCS Spike Compound	Percent Recovery Range 5 gram soil
1,1-Dichloroethene	75-138
Trichloroethene	75-121
Benzene	75-129
Toluene	76-119
Chlorobenzene	78-122

Note:

This table is subject to change without notice.

9.4.3.1 Gases and known poor purging compounds

- gases: bromomethane
  chloromethane
  chloroethane
  vinyl chloride

dichlorodifluoromethane
trichlorofluoromethane)

- acetone
- 2-butanone
- carbon disulfide
- crotonaldehyde
- 1,2-dibromo-3-chloropropane
- 1,4-dioxane
- isobutyl alcohol
- 2-hexanone
- 4-methyl-2-pentanone
- vinyl acetate
- 2-chloroethyl vinyl ether

9.4.3.2 When the LCS fails to meet the acceptance criteria, the entire batch associated with it must be re-prepared and reanalyzed.

For NYSASP, the matrix spike blank recovery criteria are the same as the matrix spike criteria shown below. When the matrix spike blank fails criteria it must be re-prepared and re-analyzed along with the matrix spikes. Associated samples are not required to be re-processed.

9.4.4 For SC DHEC, an expanded subset of analytes, representative of the compounds being reported, is employed and all analytes must have recovery limits within 70-130%. The analytes are:

- vinyl chloride
- 1,1-dichloroethene
- methylene chloride
- 1,1-dichloroethane
- cis-1,2-dichloroethene
- 2-butanone
- carbon tetrachloride
- benzene
- trichloroethene
- 1,2-dichloropropane
- bromodichloromethane
- tetrachloroethene
- chlorobenzene

- ethylbenzene
- styrene
- bromoform
- 1,4-dichlorobenzene

9.5 Matrix Spikes

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared and analyzed with every SDG. The MS/MSD and LCS are spiked with the same analytes. For SC DHEC the MS/MSD must also be analyzed every 20 soil samples.

9.5.2 For the MS/MSD, in addition to spiking internal standard solution and surrogate solution, also add 5.0 µl of 8260 spike solution. For a full LCS requirement, use the ICV standard. The same ICV standard can be used for projects submitted to meet the regulatory requirements of the State of South Carolina, but only the 17 analytes presented in 9.4.4 are assessed. The spiking solutions are added by piercing the septum with the syringe needle.

9.5.3 Matrix spikes have the following advisory recovery criteria as shown in Table 6A.

Spike Compound	% Recovery Range
1,1-dichloroethene	59-172
trichloroethene	62-137
Benzene	66-142
toluene	59-139
chlorobenzene	78-122
All others	50-150

Note: This table is subject to change without notice.

9.5.4 Matrix spikes have the following advisory RPD criteria as shown in Table 6B.

Spike Compound	% RPD
1,1-dichloroethene	22

trichloroethene	24
benzene	21
toluene	21
chlorobenzene	21
All others	25

Note: This table is subject to change without notice.

9.5.5 Most spike compounds should meet these criteria. If the criteria are not met in the MS/MSD but are met in the LCS, the results may be reported with the failures attributed to the matrix of the sample. If the LCS does not meet criteria, then all will have to be repeated as discussed above.

9.6 Duplicates

9.6.1 Duplicates, at a frequency of 5%, are required when processing soil samples submitted to meet the regulatory requirements of South Carolina DHEC. This can be satisfied with the MS/MSD.

9.7 Initial Calibration Verification

9.7.1 A second source initial calibration verification (ICV) standard is run after the initial calibration standards have met criteria.

9.7.2 The ICV must be within 20% of its expected value for each target analyte and surrogate or within 40% for the poor purgers and the gases. Sporadic failure of up to three target compounds is allowed but they must not exceed 40% of their expected value. Gases and poor purgers are listed above.

9.8 MDL Check Samples

9.8.1 On an annual basis a method detection limit (MDL) study is performed on a single instrument per method and matrix. When multiple instruments are used for the analysis of U.S. Army Corps of Engineers samples (or samples from other programs with the same requirement), individual instrument MDL studies are replaced by the analysis of an MDL check sample. The MDL check sample must be analyzed on all instruments, where the MDL study was not performed and that U.S. Army Corps of Engineers samples (or other program-specific samples) are analyzed on, to demonstrate equivalent sensitivity.

The MDL check sample is prepared at about 2x the MDL and is analyzed on a quarterly basis for each matrix. A response must be detected in the

2x MDL check sample. Additionally, qualifying ions of 50% or higher must also be present. For more information on MDL studies, refer to QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies."

10.0 Calibration and Standardization

10.1 BFB Tuning

10.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2ul STD ID#7008) into the GC using a 10-µl Hamilton syringe. BFB may be analyzed simultaneously with a continuing calibration verification standard as long as all QC criteria are met.

10.1.2 The peak selection criteria for BFB analysis are as follows (in order of preference):

10.1.2.1 Average one scan prior to the apex of the BFB peak to one scan after the apex, subtracting a single background scan prior to the peak, but no more than 20 scans prior to the elution of BFB. Also, do not subtract part of the BFB peak.

Note: For work performed to comply with the requirements of the NYSASP, only this option is allowed.

10.1.2.2 Choose the apex of the BFB peak only and include background subtraction. *

* Background subtraction is performed to eliminate interference and when performed, the subtracted scan must be no more than 20 scans prior to the elution of the BFB and no scans within the BFB peak may be subtracted.

10.1.2.3 Choose a single scan or a range of scans within the BFB peak and include background subtraction.*

Note: The use of a single scan other than at the apex is not allowed for Army Corps of Engineers project work.

10.1.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in Table 7.

Table 7: BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 - 40% of M/Z 95
75	30 - 60% of M/Z 95
95	Base Peak; 100% relative abundance
96	5 - 9% of M/Z 95
173	<2% of M/Z 174
174	>50% of M/Z 95
175	5 - 9% of M/Z 174
176	>95% but less than 101% of m/z 174
177	5 - 9% of M/Z 176

10.1.4 Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2) provided that method performance isn't adversely affected.

10.1.5 All criteria must be met according to requirements established by the U.S. EPA shown above. BFB technical acceptance criteria must be met before any standards, samples, or required blanks are analyzed.

GC/MS tuning and Mass Calibration forms must be printed and attached to the instrument runlog page for each tune. The relative abundance for each ion is calculated to two decimal places.

10.1.6 If BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective action to achieve the technical acceptance criteria.

10.2 Initial Calibration

10.2.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution (BFB) criteria have been met, each GC/MS system must be calibrated at six concentrations to demonstrate instrument sensitivity and the linearity of responses for the purgeable target compounds.

10.2.2 Prepare standards according to the Initial Calibration Standard Preparation Table 2 in Section 7.5. The purge and trap volume is 5 ml. All initial calibration standards must be analyzed at the concentration levels and frequency described in this SOP on a GC/MS system meeting the BFB

technical acceptance criteria. The analysis of the six calibration standards determines the linearity of the six-point initial calibration curve.

10.2.3 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard. Relative response factors (RRF) are calculated for each compound.

10.2.4 Initial calibration technical acceptance criteria must be met before any samples or required blanks are analyzed.

For analyses following the NYSASP, the initial calibration requirements, after meeting the instrument performance check (tune) requirements of this SOP, are those of the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics (for 1-5 g analyses).

10.2.5 Minimum relative response factors for the System Performance Check Compounds (SPCCs) are listed in Table 8.

Table 8: Relative Response Factor Criteria for SPCCs

Volatile Compound	Minimum RRF
Chloromethane	0.10
1,1-dichloroethane	0.10
chlorobenzene	0.30
bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

10.2.6 The %RSD for each target analyte should be less than 15%, but the following compounds have maximum %RSD criteria of 30%: These Calibration Check Compounds (CCC) include:

- vinyl chloride
- 1,1-dichloroethene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene

10.2.6.1 If the %RSD is 15% or less, the average relative response factor may be used for quantitation. If the %RSD is greater than 15%

then an alternate method for quantitation, such as a linear calibration using least squares regression or a non-linear calibration method, such as quadratic fit, may be used. When one of these options is used, the line must not be forced through the origin (zero) and the correlation coefficient of the equation must be 0.99 or greater for a valid calibration. If a quadratic equation is used, six levels of standards must be employed. The alternate method of quantitation is available in the ThruPut system.

For samples submitted to meet the regulatory requirements of the State of South Carolina, the option of using a quadratic fit to demonstrate linearity is not allowed. However, the use of a linear regression analysis for each target analyte is allowed.

10.2.6.2 Because of the large number of target analytes, some of them may exceed the 15% criteria. When this occurs, certain steps may be performed. These corrective actions also pertain to those instances where the action limits, or client/project specified maximum limits, have been exceeded by the non-criteria compounds.

- Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the column, service the Archon autosampler, or the purge and trap concentrator, or take other corrective action to achieve the technical acceptance criteria.

- Compare responses for the analyte in each of the standard levels to verify that a single standard analysis is not producing the outliers. If so, reanalyze that standard and recalculate the %RSD.

- The calibration range may be narrowed to determine if linearity can be achieved. This may cause more dilution re-analyses or even change the reporting limit if the lower standard is eliminated. For this method, the method quantitation limit is defined by the lowest standard.

10.2.7 The initial calibration may still be acceptable when some analytes exceed the 15% RSD criteria, if the following conditions are met and allowed by the client:

- The mean of all %RSD values for the analytes (grand mean) is less than or equal to 15%.

- All analytes in calibration the standard must be included in the calculation.

- Non-CCC target compounds have a warning limit of 50% RSD and an action limit of 90% RSD when the "grand mean" approach is used. These have been inserted as default values into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.2.7.1 A summary of the initial calibration data and/or a list of the analytes not meeting the 15% RSD criteria and the actual %RSD for each of these analytes must be included as a deliverable to our client. If the conditions in 10.2.7 are met, then the average relative response factor may be used to determine the concentration of analytes in samples.

10.2.7.2 For samples submitted to meet the regulatory requirements of the State of South Carolina, the grand mean option is not allowed, therefore, the linear regression analysis calibration model is used.

10.2.8 The initial calibration verification must be analyzed after each initial calibration and must meet the acceptance criteria. The ICV establishes the validity of the curve. If the ICV fails, then a new initial calibration curve must be generated.

10.3 Continuing Calibration Verification

10.3.1 Before the analysis of samples and blanks, but after BFB and initial calibration acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration verification standard. This standard contains all purgeable target analytes and surrogate compounds. It is used to ensure that the instrument meets the

sensitivity and linearity requirements of the method throughout the analytical sequence.

10.3.2 A check of the calibration curve must be performed once every 12 hours, beginning with the injection of BFB. A percent difference of the response for each compound compared to the mean relative response factor from the initial calibration is calculated when performing the average response factor model.

10.3.3 The calculated percent difference must be less than or equal to 20% for the CCCs listed above in Section 10.2.6. Minimum response factor criteria for the continuing calibration verification standard are also shown above in Table 8.

10.3.3.1 If a regression fit model was used for analytes in the initial calibration, the continuing calibration verification is performed using percent drift (difference) for the CCCs.

10.3.4 As indicated for the initial calibration acceptance criteria, for the continuing calibration verification, the remaining target analytes (non-CCC compounds) do not have defined % difference criteria. We have established a warning limit of 50%D and an action limit of 90%D. These values have been inserted as defaults into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD results in rejection of data.

10.3.4.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, the non-CCC target analytes should be less than 50%.

10.3.5 If continuing calibration verification acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration will have to be performed.

Note: Method 8260B indicates that if the CCCs are not required analytes, then all required analytes, must meet the 20% difference/drift criterion. Our typical analysis includes all of the CCCs. Additionally, some programs may require all compounds to meet a % difference criteria. In these situations, if the average of the response for all analytes is within 20%, then the calibration has been verified. Requirements similar to those in 10.2.6 must be met.

10.3.6 For analyses following the NYSASP, the continuing calibration requirements, after meeting the instrument performance check (tune) requirements of this SOP, are those of the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics (for 1-5g analyses).

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. All injections must be recorded on the instrument runlog (Attachment 4) along with the date, time (use a 24 hour clock), the volume injected, operator ID, and any comments relevant to the injection.

All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.1 Instrument Software Conventions

11.1.1 Quantitation method: Average of the whole

11.1.2 File naming convention: XX123456X78

where: XX = Analysis type
123456 = Date/Lab ID with first digit dropped
Y = shift
78 = instrument #

11.1.3 Analysis type prefixes

11.1.3.1 Standard: CS, CT, CU, CV, CW, CX

11.1.3.2 Initial Sample Injection:

- SDG-Sample Number-Shift-Instrument,
  e.g. Q1636-1A52

11.1.3.3 Sample reinjection:

- SDG-Sample Number-J-Shift-Instrument,
  e.g. Q1636-1JA52

11.1.3.4 Sample reextraction:

- SDG-Sample Number-R-Shift-Instrument,
  e.g. Q1636-1RA52

11.1.3.5 Sample dilution:

- SDG-Sample Number-D-Shift-Instrument, e.g Q1636-1DA60

11.1.3.6 Additional repeats:

- SDG-Sample Number-(J2, R2, D2)-Shift-Instrument, e.g. Q1636-1J2A52

11.2 Analytical Sequence

11.2.1 Order of analysis for the instrument calibration

- BFB (tune)
- initial calibration
- initial calibration verification

11.2.2 Order of analysis for the twelve-hour tune

- BFB
- continuing calibration verification
- instrument blank
- laboratory control sample-LCS
- samples

11.2.3 In some cases, if tune time remains after the initial calibration standards have been run, samples may be analyzed as long as they are preceded by a valid instrument blank.

11.2.4 All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.3 Preparations

11.3.1 Standards

11.3.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2ul Standard ID#7008) into the GC using a 10-µl Hamilton syringe.

11.3.1.2 Calibration standards are prepared by spiking the appropriate volume of each standard solution into 5 mL of sparged DI water contained in a 5 mL syringe. This is then added to a 40 mL vial containing a magnetic stirring bar and 1g of sodium bisulfate. Each vial is immediately capped with a PTFE-lined, septum-sealed cap and loaded into the Archon autosampler.

Initiate the Archon autosampler which will provide stirring, the addition of 5 mL of water containing 1 µl of internal standards and 1 ul of surrogate (5 ul of 50 ug/ml solutions if spiked by the chemist and not the Archon), heating at 40°C, and purging for 11 minutes. The system will then transfer the constituents in the headspace to the Tekmar 3000 purge and trap concentrator and will then desorb all target analytes for 4 minutes before analysis. The analyses of the six calibration standards determine the linearity of the six-point initial calibration curve.

11.3.2 Instrument Blank and Method Blank

11.3.2.1 An instrument blank is prepared by filling a 40 mL VOA vial, containing a stir bar and 1g of sodium bisulfate, with 5 mL of purged DI water and sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, 1 µl of internal standards, and 1 µl of surrogates are added automatically to the blank (5 ul of 50 ug/ml solutions if spiked by the chemist and not the Archon). It is analyzed by a closed system heated purge and trap analysis.

11.3.2.2 A Method Blank is similar to an Instrument Blank in composition but it is prepared at the same time samples are prepared and is stored in the refrigerator. If samples are received from the field already in vials with the sodium bisulfate preservative solution, only an Instrument Blank is required.

11.3.3 Laboratory Control Sample

11.3.3.1 A laboratory control sample (LCS) is prepared by filling a 40 mL VOA vial, containing a stir bar and 1g of sodium bisulfate, with 5 mL of purged DI water. To this 10 µl of the spiking standard is added before sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, 1 µl of internal standards, and 1 µl of surrogates are added

automatically (5 ul of 50 ug/ml solutions if spiked by the chemist and not the Archon). It is analyzed by a closed system heated purge and trap analysis.

11.3.3.2 For certain projects and programs, a full list spike is required.

11.3.4 Samples

11.3.4.1 Solid samples are prepared by Method 5035. For details see Sample Preparation Procedure –238: "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap." This results in 5g samples being stored in separate sealed 40 mL VOA vials containing sample, a stirring bar, and a sodium bisulfate aqueous solution (0.2g sodium bisulfate per g of sample).

11.3.4.2 The choice of whether a 5g or medium level sample is analyzed is generally based on a screen analysis.

11.3.4.3 Samples are stored in a rack located in the volatile GC/MS laboratory refrigerator at 2°C to 4.4°C. Samples are allowed to come to room temperature and then loaded into the Archon autosampler carousel shaking each vial gently so that the contents move freely and the stirring bar will be able to spin.

11.3.5 Matrix Spikes

11.3.5.1 For sample spikes, the Archon will spike 1 µl of internal standard solution and 1 µl of surrogate solution (5 ul of 50 ug/ml solutions if spiked by the chemist and not the Archon), also add 10.0 µl of 8260B spike solution.

11.3.5.2 For certain projects, full target list matrix spikes are required.

11.4 Analysis

11.4.1 When the Archon autosampler is initiated, the system will add 5 mL of purged DI water, containing 5 µl of internal standards and 5 µl of surrogates, by piercing the septum.

11.4.2 Prior to purging, the stirring bar is turned on and the sample is heated to 40°C. The sample is purged for 11 minutes, while stirring.

11.4.3 The same needle that is used to add the DI water is the source for the inert gas used for purging. The needle also contains slots above the sample/water level which provide a path for the headspace to be directed to the Tekmar 3000 purge and trap concentrator. This contains the trap that is then thermally desorbed into the GC/MS instrument.

11.4.4 After purging, the Purge and Trap Concentrator apparatus will desorb onto the GC column by elevating the trap temperature to 260°C and back-flushing the trap with helium for 4 minutes at 20 to 60 mL/minute.

11.4.5 After desorbing, the trap is reconditioned by baking at 260°C for at least 7 minutes. When the trap has finished baking and is cool, it is ready for the next sample to be purged.

11.4.6 In each analytical run, all analytes must fall below the maximum calibration range established by the highest standard in the initial calibration. If an analyte is present at a concentration higher than the highest initial calibration standard, it must be reanalyzed at a lesser amount or dilution. A valid dilution is one in which the compound in question falls above the mid-point calibration standard concentration. The dilution is considered valid if the analyte concentration is above 50 µg/kg.

11.5 Identification

11.5.1 Target compounds are identified in the samples by analyzing standards under the same conditions used for samples. The resulting mass spectra are compared to established library spectra and GC retention times to retention times from the latest continuing calibration standard. The mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria:

11.5.1.1 All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.

11.5.1.2 The relative intensities of ions specified above must agree within ±20% between the library and sample spectra.

11.5.1.3 Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for.

11.5.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral

interpretation specialist the identification is correct, then the laboratory will report that identification.

11.5.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library.

11.6 Quantitation

11.6.1 The mean relative response factor (RRF) from the initial calibration standard is used to calculate the concentration in the sample. For NYSASP, the RRF from the continuing calibration standard is used to calculate concentrations.

Note: Alternatively, the calibration curve(s) generated from the initial calibration may be used for the determination of analyte(s) concentration(s). This option is discussed above.

11.6.2 All samples require a search of all extraneous peaks >10% of the height of the nearest internal standard, up to 10 searches, i.e. 10 most intense extraneous peaks. The number of searches may be more, depending on client requirements.

11.6.3 In each analytical run, all analytes must fall below the method's maximum analytical range, i.e. the highest calibration standard.

11.6.3.1 If an analyte is present at a concentration higher than the maximum analytical range in a 5g analysis, the medium level sample must be analyzed.

11.6.4 When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by the analysis of an instrument blank or the following sample must be monitored for contamination and interference from carryover. If the blank or sample is not free from interferences, the system must be decontaminated. Sample analysis may not resume until a blank or sample has been analyzed which is free from interferences. Being free from interferences means that whatever compound was present above the initial calibration range in a sample, cannot be present in an instrument blank or the sample analyzed immediately following, at a level above the reporting limit for that compound.

11.6.5 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative.

11.6.6 Non-target compounds are quantified by comparing the MS response from the reconstructed ion chromatogram (RIC) for the non-target compound peaks to the MS response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ \mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of %RSD

$$\%\text{RSD} = \left(\frac{\textit{Standard deviation}}{\overline{\text{X}}} \right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \times 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax \times C(is)}{A(is) \times Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured
A(is) = Area of the characteristic ion (EICP) for the specific internal standard
C(is) = Concentration of the internal standard (in µg/l)
Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.9 Concentration

12.9.1 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard.

12.9.2 Concentration of soil samples (dry weight basis) by GC/MS analysis using relative response factor:

$$ug/kg = \frac{(As)(Cis)}{(Ais)(\overline{RF})(Ws)(D)}$$

where: As = Area of the peak for the analyte in the sample
Ais = Area of the peak for the internal standard
Cis = Concentration of the internal standard in the volume purged, in µg/L
$\overline{RF}$ = Mean response factor from the initial calibration

$$\overline{RF} = \frac{\sum_{i=1}^{n} RFi}{n}$$

Ws = weight of sample purged, in grams

$$D\ (dry\ weight) = \frac{100 - \%\ moisture}{100}$$

12.9.3 Concentration of soil samples (dry weight basis) by GC/MS using quadratic (second order) fit in Target:

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where: b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
n = amount of Internal Standard
y = concentration in ng on column
Rsp = area of analyte/area of Internal Standard

Example: Area of acetone = 35659
Area of IS = 613275
b = -0.0909161
m^1 = 9.605304
m_2 = 7.132688
ng of IS = 250
response = 35659/613275 = 0.058145

Amount in ng on column =

$$(250ng)[-0.0909161 + 9.605304\ x\ 0.058145 + 7.132688\ x\ 0.058145\text{^}2] = 122.9ng$$

$$Concentration\ \mu g / Kg = \frac{122.9ng}{(Ws)(D)}$$

12.9.4 Concentration of soil samples (dry weight basis) by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where: As = Area of the target analyte peak in the sample
Ais = Area of the internal standard peak
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

$$Concentration\ \mu g / Kg = \frac{(Cs)}{\mathbf{(Ws)}(D)}$$

12.9.5 Tentatively Identified Compound (TIC) Estimation

$$TIC\ Amount = \frac{(Area\ TIC)\ x\ Amount(Std)}{(Area\ IS)\ x\ 1(RF)(Ws)(D)}$$

where: Area (TIC) = area response from RIC for non-target compound
Amount(Std) = amount of internal standard added to the sample, in µg/L.
Area (IS) = area response of the nearest internal standard in the reconstructed ion chromatogram

1(RF) = assumed response factor of 1

12.10 Calculating Dilutions

12.10.1 If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution.

Adjust the amount of sample purged with 1 gram being the lowest acceptable weight for a low level analysis. If the analyte still exceeds the analytical range in the medium level analysis, perform a methanol extraction following Sample Preparation Procedure –238, "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment 3) and precision and accuracy for single analyst (Attachment 6). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and

controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. U.S. EPA SW846 3rd Edition, Update 3, 12/96, Methods 8260B and 5035

16.2 New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 QCSOP: Proper Documentation Procedures

16.5 QCSOP: Numerical Data Reduction

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, July 2002, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.11 CompuChem Quality Manual, Revision **6**, **11**/**24**/04, plus revisions

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 Sample Preparation Procedure –238, "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Target Compound List

17.2 Attachment 2 – Statistical Control Limits

17.3 Attachment 3 - Method Detection Limits

17.4 Attachment 4 – Instrument Runlog

17.5 Attachment 5 – Standard Certificates of Analysis

17.6 Attachment 6 – Single Analyst Capability Study

Attachment 1

Table 1: Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
dichlorodifluoromethane	1	85	87
chloromethane	1	50	52
vinyl chloride	1	62	64
bromomethane	1	94	96
chloroethane	1	64	66
trichlorofluoromethane	1	101	103
1,1-dichloroethene	1	96	61, 98
methylene chloride	1	84	49, 86
trans-1,2-dichloroethene	1	96	61, 98
1,1-dichloroethane	1	63	65, 83
2,2-dichloropropane	1	77	97
cis-1,2-dichloroethene	1	96	61, 98
bromochloromethane	1	128	49, 130
chloroform	1	83	85
1,1,1-trichloroethane	1	97	99, 61
carbon tetrachloride	1	117	119, 121
1,1-dichloropropene	1	75	110, 77
benzene	1	78	77, 51
1,2-dichloroethane	1	62	98
trichloroethene	1	130	95, 97
1,2-dichloropropane	1	63	112
dibromomethane	1	174	93, 95
bromodichloromethane	1	83	85, 127
2-chloroethyl vinyl ether	1	63	65, 106
cis-1,3-dichloropropene	1	75	77
acrolein	1	56	55, 58
iodomethane	1	142	127, 141
1,1,1-trichloro-2,2,2,-trifluoroethane	1	117	151, 153
1,1,2-trichloro-1,2,2,-trifluoroethane	1	85	101, 151
carbon disulfide	1	76	78
acetone	1	43	58
3-chloropropene	1	76	41, 78
acetonitrile	1	41	40, 39

NOTE: Attachment is subject to change without notice.

Attachment 1 (continued)

Table 1 (continued): Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
Acrylonitrile	1	53	52, 51
methyl-tert-butyl ether	1	73	41, 43
vinyl acetate	1	43	86
2-butanone	1	72	43, 57
Propionitrile	1	54	55, 52
Methacrylonitrile	1	41	39, 67
1-chlorobutane	1	56	49
1,4-dioxane	1	88	58
Methylmethacrylate	1	69	100, 41
Surrogate #1: Dibromofluoromethane	1	113	111, 192
Surrogate #2: d4-1,2-dichloroethane	1	65	102, 67
4-methyl-2-pentanone	2	43	85, 100
Toluene	2	92	91
trans-1,3-dichloropropene	2	75	77
1,1,2-trichloroethane	2	97	83, 85
Ethylmethacrylate	2	69	41, 99
Tetrachloroethene	2	164	168, 129
1,3-dichloropropane	2	76	78
2-hexanone	2	43	58, 57
Dibromochloromethane	2	129	127, 48
1,2-dibromoethane	2	107	109, 188
Chlorobenzene	2	112	114, 77
1,1,1,2-tetrachloroethane	2	131	119, 133
Ethylbenzene	2	106	91
m,p-xylene	2	106	91
o-xylene	2	106	91
Styrene	2	104	91, 78
Bromoform	2	173	175, 254
isopropyl benzene	2	105	120
Bromobenzene	2	156	77,158
1,1,2,2-tetrachloroethane	2	83	85, 131

NOTE: Attachment is subject to change without notice.

Attachment 1 (continued)

Table 1 (continued): Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
1,2,3-trichloropropane	2	110	75, 112
trans-1,4-dichloro-2-butene	2	53	88, 75
Surrogate #3: d8-toluene	2	98	70, 100
n-propyl benzene	3	91	120
2-chlorotoluene	3	126	91
4-chlorotoluene	3	91	126
1,2,4-trimethyl benzene	3	105	120
1,3,5-trimethyl benzene	3	105	120
Pentachloroethane	3	167	130, 165
sec-butyl benzene	3	105	134
1,2-dichlorobenzene	3	146	111, 148
1,3-dichlorobenzene	3	146	111, 148
1,4-dichlorobenzene	3	146	111, 148
n-butyl benzene	3	91	92, 134
tert-butyl benzene	3	119	91, 134
p-isopropyl toluene	3	119	134, 91
1,2-dibromo-3-chloropropane	3	75	155, 157
1,2,4-trichlorobenzene	3	180	182, 145
Hexachlorobutadiene	3	225	223, 227
Naphthalene	3	128	64, 51
1,2,3-trichlorobenzene	3	180	182, 145
Surrogate #4: 4-bromofluorobenzene	3	95	174, 176
Internal Standard #1: fluorobenzene	NA	96	70
Internal Standard #2: d5-chlorobenzene	NA	117	82, 119
Internal Standard #3: d4-1,4-dichlorobenzene	NA	152	150

[1] Based on laboratory tests, 2-chloroethyl vinyl ether is not analyzable from the sodium bisulfate solution associated with Method 5035.

Note: Attachment is subject to change without notice.

Attachment 2

Table 2: Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Dichlorodifluoromethane	50-142
Chloromethane	52-150
Vinyl chloride [2]	58-148
Bromomethane	50-150
Chloroethane	52-150
Trichlorofluoromethane	54-144
Diethyl ether	50-149
Acrolein	50-150
1,1-dichloroethene [1, 2]	51-146
Iodomethane	50-150
1,1,1-trichloro-2,2,2,-trifluoroethane	58-148
Carbon disulfide	56-140
1,1,2-trichloro-1,2,2,-trifluoroethane	50-149
Acetone	50-147
3-chloropropene	50-150
Acetonitrile	68-126
Methyl acetate	50-150
Methylene chloride [2]	50-137
trans-1,2-dichloroethene	65-122
Acrylonitrile	50-136
Methyl-tert-butyl ether	70-133
Tert butyl alcohol	60-148
n-hexane	50-143
1,1-dichloroethane [2]	66-123
Chloroprene	50-150
Vinyl acetate	50-150
Isopropyl ether	81-114
2,2-dichloropropane	64-141
cis-1,2-dichloroethene [2]	69-127
2-butanone [2]	66-132
Propionitrile	50-150
Bromochloromethane	73-126
Methyl acrylate	50-133
Methacrylonitrile	51-127
Tetrahydrofuran	50-150

Note: Attachment is subject to change without notice.

Attachment 2 (continued)

Table 2: Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Chloroform	75-128
1,1,1-trichloroethane	71-127
Cyclohexane	65-126
1-chlorobutane	75-124
Carbon tetrachloride [2]	67-132
1,1-dichloropropene	72-132
Pentafluorobenzene	81-117
Benzene [1, 2]	67-126
1,2-dichloroethane	56-141
Isobutyl alcohol	50-150
Crotonaldehyde	50-141
Trichloroethene [1, 2]	69-130
Methylcyclohexane	80-119
1,2-dichloropropane [2]	73-121
Dibromomethane	64-131
1,4-dioxane	50-150
Methylmethacrylate	60-127
Bromodichloromethane [2]	75-133
2-chloroethyl vinyl ether	50-150
cis-1,3-dichloropropene	87-127
4-methyl-2-pentanone	67-125
Toluene [1]	73-121
trans-1,3-dichloropropene	64-131
1,1,2-trichloroethane	67-123
Ethylmethacrylate	67-124
Tetrachloroethene [2]	72-130
1,3-dichloropropane	75-130
2-hexanone	51-128
Dibromochloromethane	68-127
1,2-dibromoethane	69-127
Chlorobenzene [1, 2]	71-120
1-chlorohexane	80-113
1,1,1,2-tetrachloroethane	82-120
Ethylbenzene [2]	76-121
m,p-xylene	76-130

Note: Attachment is subject to change without notice.

Attachment 2 (continued)

Table 2: Statistical Control Limits for the LCS

Compounds	Percent Recovery Range 5 gram soil
o-xylene	76-130
Styrene [2]	68-126
Bromoform [2]	64-134
Isopropyl benzene	68-126
cis-1,4-dichloro-2-butene	50-150
Bromobenzene	67-140
1,2,3-trichloropropane	58-137
1,1,2,2-tetrachloroethane	66-128
trans-1,4-dichloro-2-butene	50-150
n-propyl benzene	71-136
2-chlorotoluene	78-124
4-chlorotoluene	74-131
1,3,5-trimethyl benzene	72-129
Pentachloroethane	64-150
tert-butyl benzene	67-140
1,2,4-trimethyl benzene	70-133
sec-butyl benzene	67-141
1,3-dichlorobenzene	74-119
1,4-dichlorobenzene [2]	72-115
p-isopropyl toluene	74-132
Benzyl chloride	63-124
1,2-dichlorobenzene	67-126
n-butyl benzene	63-137
1,2-diethylbenzene	75-117
1,2-dibromo-3-chloropropane	72-127
1,2,4-trichlorobenzene	57-133
Hexachlorobutadiene	68-129
Naphthalene	50-139
1,2,3-trichlorobenzene	63-129
Xylene (total)	76-130

Table displays statistical control limits calculated in 2002.

[1] Denotes component of minimum LCS spike

[2] Denotes component of LCS spike for SC DHEC. Each of these analytes must be recovered within 70-130%.

Note: Attachment is subject to change without notice.

Attachment 3

CompuChem Method Detection Limit Study

Study date: January 8, 21, 2004		GCMS Volatile SW846 8260B/5030B/5035 Soil, 5 gm Purge without Sodium Bisulfate												
Instrument: 5972hp59														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Mean	Test Conc.	SDev	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
Dichlorodifluoromethane	5.70	5.49	5.60	5.25	5.40	5.45	5.56	5.30	5.47	2.5	0.15	0.45	5
Chloromethane	5.32	5.33	5.23	5.09	5.71	5.26	5.54	5.55	5.38	2.5	0.20	0.61	5
Vinyl Chloride	5.26	4.95	5.11	4.91	5.21	5.05	5.54	5.01	5.13	2.5	0.20	0.61	5
Bromomethane	4.70	4.58	4.43	4.43	4.87	4.19	4.91	3.80	4.49	2.5	0.37	1.10	5
Chloroethane	5.52	4.76	5.07	4.80	4.92	4.98	5.04	5.42	5.06	2.5	0.27	0.82	5
Trichlorofluoromethane	4.18	4.13	3.97	4.65	4.80	4.74	4.94	4.91	4.54	2.5	0.39	1.16	5
Acrolein	26.65	30.52	27.68	25.19	27.44	27.12	26.27	24.24	26.9	25	1.87	5.61	50
1,1-Dichloroethene	2.80	2.94	2.95	2.89	2.97	2.92	2.61	2.88	2.87	2.5	0.12	0.35	5
1,1,1-Trichloro-2,2,2-trifluoroethane	2.33	2.32	2.46	2.72	3.00	2.97	2.75	2.96	2.69	2.5	0.29	0.86	5
Acetone	12.43	12.78	13.67	11.85	10.59	12.07	11.92	12.30	12.2	5.0	0.88	2.63	13
Iodomethane	2.31	2.48	2.42	2.70	2.61	2.54	2.69	2.43	2.52	2.5	0.14	0.41	5
1,1,2-trichloro-1,2,2-trifluoroethane	2.55	2.67	2.73	3.08	3.09	3.18	2.98	2.89	2.90	2.5	0.23	0.68	5
Carbon disulfide	1.94	1.80	1.91	1.75	1.79	1.60	1.91	1.83	1.82	2.0	0.11	0.33	5
3-Chloropropene	0.47	0.65	0.66	0.44	0.80	0.37	1.63	0.59	0.70	2.5	0.40	1.20	5
Acetonitrile	3.79	3.98	3.82	3.49	3.46	3.29	3.10	3.04	3.50	2.5	0.35	1.03	5
Methyl acetate	3.72	3.76	3.20	3.22	3.40	2.96	2.68	3.01	3.24	2.0	0.37	1.12	5
Methylene Chloride	4.52	3.90	4.26	4.43	4.19	4.43	4.43	4.28	4.31	2.5	0.20	0.59	5
Acrylonitrile	28.38	29.31	31.78	30.60	29.62	28.68	26.91	26.37	29.0	25	1.79	5.38	50
trans-1,2-Dichloroethene	2.56	2.60	2.59	2.76	2.55	2.65	2.63	2.28	2.58	2.5	0.14	0.41	5
Methyl-tert-butyl-ether	2.58	2.43	2.44	2.56	2.51	2.65	2.59	2.35	2.51	2.5	0.10	0.30	5
1,1-Dichloroethane	2.69	2.71	2.92	2.73	2.71	3.04	2.82	2.68	2.79	2.5	0.13	0.39	5
Chloroprene	2.34	2.29	2.40	2.50	2.50	2.36	2.31	2.18	2.36	2.5	0.11	0.32	5
Vinyl acetate	2.53	2.56	2.55	2.87	3.37	2.79	2.73	2.42	2.73	2.5	0.30	0.90	5
Isopropyl ether	2.44	2.32	2.32	2.93	2.96	2.26	2.26	2.04	2.44	2.5	0.33	0.99	5
cis-1,2-Dichloroethene	2.13	2.06	2.20	2.20	2.25	2.07	1.82	1.95	2.09	2.5	0.14	0.43	5
2,2'-Dichloropropane	2.68	2.49	2.63	2.67	2.59	2.41	2.50	2.29	2.53	2.5	0.14	0.41	5
Propionitrile	151.4	148.5	155.9	150.2	150.0	134.2	133.1	122.5	143	125	11.74	35.20	250
2-Butanone	9.54	7.51	8.59	8.21	9.65	7.29	7.10	6.25	8.02	6.3	1.20	3.60	13
Methacrylonitrile	7.99	8.23	7.67	8.16	6.90	6.40	6.99	5.71	7.3	20	0.91	2.73	50
Bromochloromethane	2.50	2.46	2.46	2.52	2.25	2.47	2.29	2.41	2.42	2.5	0.10	0.30	5
Chloroform	1.82	1.82	1.98	1.97	1.97	1.92	2.00	1.77	1.91	2.0	0.09	0.27	5
1,1,1-Trichloroethane	2.59	2.79	2.83	2.85	2.81	2.65	2.77	2.68	2.75	2.5	0.09	0.28	5
Cyclohexane	2.17	1.89	2.27	1.92	2.28	2.08	1.89	1.92	2.05	2.5	0.17	0.51	5
1,1-Dichloropropene	2.15	2.20	2.27	2.15	1.99	2.03	1.92	2.15	2.11	2.5	0.12	0.35	5
Carbon tetrachloride	2.55	2.55	2.88	2.66	2.72	2.79	2.73	2.67	2.69	2.5	0.11	0.34	5
1,2-Dichloroethane	1.85	1.78	1.94	1.97	1.93	1.82	1.97	1.83	1.89	2.0	0.07	0.22	5
Isobutyl alcohol	127.6	126.7	138.1	128.0	144.2	116.5	102.2	99.39	123	125	15.89	47.63	250
Benzene	1.65	1.48	1.62	1.48	1.46	1.52	1.59	1.46	1.53	2.0	0.08	0.23	5
Trichloroethene	1.63	1.31	1.57	1.37	1.11	1.22	1.16	1.23	1.33	2.0	0.19	0.57	5
Methylcyclohexane	2.01	1.79	1.84	1.61	2.18	2.42	1.46	1.47	1.85	2.5	0.34	1.02	5
1,2-Dichloropropane	2.86	2.56	2.55	2.61	2.53	2.55	2.49	2.60	2.59	2.5	0.11	0.34	5
Dibromomethane	2.31	2.28	2.57	2.24	2.23	2.36	2.11	2.16	2.28	2.5	0.14	0.42	5
1,4-Dioxane	110.64	117.11	130.17	88.09	122.1	97.74	100.4	79.59	106	125	17.35	52.01	250
Methylmethacrylate	25.50	24.92	28.42	26.22	25.76	25.90	24.44	23.27	25.6	25	1.50	4.49	50
Bromodichloromethane	2.54	2.77	2.80	2.54	2.68	2.66	2.75	2.66	2.68	2.5	0.10	0.29	5
2-Chloroethyl vinyl ether	1.51	1.31	1.71	1.07	0.83	0.91	0.89	0.45	1.09	2.5	0.41	1.22	5
cis-1,3-Dichloropropene	2.12	1.94	1.95	1.79	1.88	1.77	1.80	1.65	1.86	2.5	0.14	0.43	5

Reporting Limit = Low Level Standard

NOTE: Attachment is subject to change without notice.
NOTE: The estimated detection limit is 1/5 the reporting limit.

Attachment 3 (continued)

CompuChem Method Detection Limit Study

Study date: January 8, 21, 2004		GCMS Volatile SW846 8260B/5030B/5035 Soil, 5 gm Purge without Sodium Bisulfate												
Instrument: 5972hp59														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Mean	Test Conc.	SDev	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
4-Methyl-2-pentanone	6.34	5.52	6.01	5.31	4.78	4.92	4.35	4.24	5.18	6.3	0.75	2.26	13
Toluene	2.57	2.47	2.48	2.29	2.19	2.66	2.54	2.52	2.47	2.5	0.15	0.46	5
trans-1,3-Dichloropropene	2.94	2.82	2.78	2.91	2.72	3.00	2.48	2.60	2.78	2.5	0.18	0.53	5
1,1,2-Trichloroethane	2.66	2.70	2.99	2.55	2.65	2.54	2.59	2.57	2.66	2.5	0.15	0.44	5
Ethylmethacrylate	26.24	26.34	26.95	24.42	24.12	25.32	23.26	21.96	24.8	25	1.71	5.12	50
1,3-Dichloropropane	2.71	2.59	2.79	2.56	2.31	2.60	2.46	2.52	2.57	2.5	0.15	0.44	5
Tetrachloroethene	2.33	2.32	2.24	2.15	2.22	2.31	2.42	2.17	2.27	2.5	0.09	0.27	5
2-Hexanone	5.77	5.62	5.84	5.28	4.66	4.10	4.39	4.15	5.0	6.3	0.73	2.20	13
Dibromochloromethane	2.62	2.67	2.86	2.56	2.49	2.58	2.34	2.36	2.56	2.5	0.17	0.51	5
1,2-Dibromoethane	2.61	2.47	2.53	2.42	2.26	2.46	2.23	2.41	2.42	2.5	0.13	0.38	5
Chlorobenzene	1.58	1.64	1.77	1.69	1.71	1.71	1.78	1.69	1.70	2.0	0.07	0.19	5
1,1,1,2-Tetrachloroethane	2.60	2.59	2.78	2.71	2.51	2.69	2.47	2.58	2.62	2.5	0.10	0.31	5
Ethylbenzene	2.00	2.02	2.21	1.85	2.05	1.87	1.98	1.92	1.99	2.5	0.11	0.34	5
m,p-Xylene	2.67	2.46	2.44	2.26	2.13	2.02	2.02	2.47	2.31	4.0	0.24	0.71	10
o-Xylene	1.87	1.81	1.88	1.62	1.74	1.79	1.70	1.64	1.76	2.5	0.10	0.30	5
Styrene	3.07	1.89	1.76	1.72	1.62	1.72	1.56	1.59	1.87	2.5	0.50	1.49	5
Bromoform	2.43	2.42	2.54	2.45	2.26	2.32	2.23	2.18	2.35	2.5	0.13	0.38	5
Isopropyl benzene	1.91	1.60	1.82	1.66	1.83	1.88	1.45	1.39	1.69	2.5	0.20	0.60	5
1,1,2,2-Tetrachloroethane	2.97	2.48	3.09	2.56	2.67	2.50	2.58	2.71	2.70	2.5	0.22	0.67	5
1,2,3-Trichloropropane	1.34	2.38	2.21	2.22	2.00	2.66	2.45	1.93	2.15	2.0	0.40	1.21	5
Bromobenzene	2.95	2.76	2.91	2.74	2.82	2.84	2.62	2.67	2.79	2.5	0.11	0.34	5
trans-1,4-Dichloro-2-butene	29.84	30.18	30.31	29.82	29.74	29.87	29.58	29.38	29.8	10	0.30	0.90	20
n-Propyl benzene	2.61	2.43	2.54	2.36	3.00	2.76	2.25	2.31	2.53	2.5	0.25	0.76	5
2-Chlorotoluene	2.22	2.14	2.10	2.15	2.20	2.18	1.99	1.79	2.10	2.5	0.14	0.43	5
4-Chlorotoluene	2.45	2.12	2.14	1.98	2.28	2.34	1.94	1.92	2.15	2.5	0.20	0.59	5
1,3,5-Trimethyl benzene	2.37	2.02	2.04	2.06	2.36	2.38	1.69	1.77	2.09	2.5	0.27	0.81	5
tert-butyl Benzene	2.05	1.56	1.74	1.56	2.01	1.98	1.44	1.21	1.69	2.5	0.30	0.91	5
1,2,4-Trimethyl benzene	2.34	2.07	1.97	2.04	2.40	2.51	1.81	1.71	2.11	2.5	0.29	0.86	5
sec-butyl Benzene	2.36	2.10	2.04	1.99	2.39	2.66	1.68	1.60	2.10	2.5	0.36	1.08	5
1,3-Dichlorobenzene	2.78	2.41	2.37	2.45	2.65	2.64	2.14	2.12	2.45	2.5	0.24	0.71	5
p-Isopropyl toluene	2.21	1.85	1.87	1.81	2.27	2.37	1.40	1.42	1.90	2.5	0.37	1.10	5
1,4-Dichlorobenzene	3.01	2.58	2.71	2.53	2.86	2.98	2.49	2.35	2.69	2.5	0.24	0.72	5
1,2-Dichlorobenzene	2.99	2.53	2.34	2.45	2.62	2.45	2.21	2.22	2.48	2.5	0.25	0.76	5
n-Butyl benzene	2.82	2.13	2.11	2.06	2.44	2.82	1.69	1.48	2.19	2.5	0.48	1.45	5
1,2-Dibromo-3-chloropropane	4.86	4.61	4.79	4.57	4.67	4.55	4.27	4.21	4.57	2.5	0.23	0.68	5
1,2,4-trichlorobenzene	1.74	1.79	1.43	1.39	1.35	1.37	1.41	1.80	1.54	2.0	0.20	0.61	5
Hexachlorobutadiene	1.57	2.21	1.74	1.84	2.41	2.47	2.61	3.43	2.29	2.0	0.60	1.78	5
Naphthalene	2.63	2.03	1.74	1.24	1.19	1.14	1.09	1.27	1.54	2.0	0.55	1.65	5
1,2,3-Trichlorobenzene	2.06	1.95	1.68	1.48	1.48	1.62	1.50	1.87	1.71	2.0	0.23	0.68	5

Reporting Limit = Low Level Standard

NOTE: Attachment is subject to change without notice.
NOTE: The estimated detection limit is 1/5 the reporting limit.

Attachment 3 (continued)

CompuChem Method Detection Limit Study

Study date: March 25, 2004		GCMS Volatile SW846 8260B/5030B/5035 Soil, 5 gm Purge with Sodium Bisulfate												
Instrument: 5972hp52														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Mean	Test Conc.	SDev	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
Dichlorodifluoromethane	0.75	1.13	1.02	1.35	1.23	1.19	1.12	1.10	1.11	2.5	0.176	0.53	5
Chloromethane	0.86	1.52	1.18	1.50	1.60	1.58	1.48	1.33	1.38	2.5	0.252	0.76	5
Vinyl Chloride	0.99	1.58	1.35	1.65	1.62	1.60	1.36	1.45	1.45	2.5	0.220	0.66	5
Bromomethane	1.01	1.46	1.21	1.46	1.47	1.33	1.32	1.30	1.32	2.5	0.156	0.47	5
Chloroethane	0.83	1.16	0.87	1.22	1.23	1.09	1.00	1.01	1.05	2.5	0.151	0.45	5
Trichlorofluoromethane	1.47	1.74	1.42	1.78	1.74	1.69	1.50	1.50	1.61	2.5	0.146	0.44	5
Acrolein	36.19	30.38	23.03	24.42	20.78	21.15	22.35	*	25.5	25	5.722	18.0	50
1,1-Dichloroethene	1.36	1.74	1.42	1.67	1.54	1.54	1.44	1.47	1.52	2.5	0.129	0.39	5
1,1,1-Trichloro-2,2,2-trifluoroethane	1.05	1.36	1.21	1.60	1.47	1.44	1.36	1.23	1.34	2.5	0.173	0.52	5
Acetone	*	15.40	10.05	13.14	15.04	11.55	16.17	21.34	14.7	5.0	3.670	11.5	13
Iodomethane	1.51	1.73	1.47	1.70	1.74	1.70	1.63	1.61	1.64	2.5	0.101	0.30	5
1,1,2-trichloro-1,2,2-trifluoroethane	1.46	1.78	1.52	1.76	1.83	1.72	1.62	1.61	1.66	2.5	0.131	0.39	5
Carbon disulfide	1.61	1.85	1.50	1.77	1.74	1.66	1.56	1.55	1.66	2.0	0.122	0.37	5
3-Chloropropene	1.07	1.35	1.02	1.35	1.28	1.23	0.96	1.11	1.17	2.5	0.152	0.45	5
Acetonitrile	3.06	3.58	2.81	3.19	3.28	3.04	3.16	3.18	3.16	2.5	0.220	0.66	5
Methyl acetate	2.82	2.66	1.84	2.12	2.08	1.75	1.53	1.47	2.03	2.0	0.494	1.48	5
Methylene Chloride	2.06	2.15	1.75	2.16	2.18	2.16	1.96	2.15	2.07	2.5	0.149	0.45	5
Acrylonitrile	26.50	23.23	16.26	20.89	20.61	21.38	19.93	21.94	21.3	25	2.905	8.71	50
trans-1,2-Dichloroethene	1.30	1.51	1.19	1.42	1.44	1.30	1.29	1.22	1.33	2.5	0.112	0.34	5
Methyl-tert-butyl-ether	1.74	1.85	1.35	1.73	1.72	1.64	1.54	1.55	1.64	2.5	0.156	0.47	5
1,1-Dichloroethane	1.53	1.71	1.39	1.66	1.66	1.58	1.49	1.54	1.57	2.5	0.105	0.31	5
Chloroprene	1.36	1.45	1.10	1.42	1.39	1.26	1.18	1.15	1.29	2.5	0.134	0.40	5
Vinyl acetate	1.69	1.81	1.37	1.64	1.52	1.38	1.29	1.30	1.48	2.5	0.160	0.48	5
Isopropyl ether	1.65	1.67	1.35	1.60	1.57	1.57	1.44	1.45	1.54	2.5	0.112	0.34	5
cis-1,2-Dichloroethene	1.37	1.48	1.18	1.37	1.37	1.38	1.25	1.30	1.34	2.5	0.092	0.28	5
2,2'-Dichloropropane	1.97	1.88	1.72	1.84	1.76	1.78	1.63	1.61	1.77	2.5	0.122	0.37	5
Propionitrile	109.25	108.61	79.14	102.61	107.56	108.21	101.57	111.34	103.5	125	10.401	31.2	250
2-Butanone	13.73	12.89	6.61	10.59	12.61	10.36	11.55	9.41	11.0	6.3	2.277	6.83	13
Methacrylonitrile	10.77	10.50	7.60	9.95	10.19	9.97	9.45	10.07	9.81	20	0.976	2.93	50
Bromochloromethane	1.32	1.38	1.08	1.42	1.47	1.32	1.18	1.23	1.30	2.5	0.130	0.39	5
Chloroform	1.49	1.56	1.25	1.52	1.60	1.51	1.37	1.50	1.48	2.0	0.113	0.34	5
1,1,1-Trichloroethane	1.48	1.52	1.25	1.57	1.52	1.49	1.37	1.29	1.44	2.5	0.118	0.35	5
Cyclohexane	1.26	1.35	1.16	1.41	1.41	1.27	1.16	1.14	1.27	2.5	0.111	0.33	5
1,1-Dichloropropene	1.27	1.33	1.21	1.33	1.29	1.26	1.19	1.17	1.26	2.5	0.061	0.18	5
Carbon tetrachloride	1.36	1.43	1.13	1.39	1.39	1.36	1.30	1.23	1.32	2.5	0.100	0.30	5
1,2-Dichloroethane	1.46	1.50	1.08	1.35	1.45	1.36	1.28	1.27	1.34	2.0	0.136	0.41	5
Isobutyl alcohol	125.03	127.52	82.14	116.66	119.69	126.52	115.49	125.26	117	125	14.920	44.7	250
Benzene	1.52	1.62	1.34	1.55	1.54	1.49	1.42	1.42	1.49	2.0	0.090	0.27	5
Trichloroethene	1.33	1.47	1.27	1.51	1.53	1.41	1.29	1.32	1.39	2.0	0.103	0.31	5
Methylcyclohexane	1.06	1.12	0.96	1.16	1.20	1.10	1.04	0.98	1.08	2.5	0.084	0.25	5
1,2-Dichloropropane	1.41	1.46	1.17	1.42	1.44	1.39	1.40	1.20	1.36	2.5	0.111	0.33	5
Dibromomethane	3.47	3.51	3.21	3.52	3.51	3.51	3.40	3.44	3.45	2.5	0.104	0.31	5
1,4-Dioxane	212.65	216.92	187.06	215.40	210.62	228.70	207.23	222.96	212.69	125	12.410	37.2	250
Methylmethacrylate	52.67	52.38	48.09	51.61	51.46	51.47	50.59	51.40	51.21	25	1.411	4.23	50
Bromodichloromethane	1.44	1.44	1.06	1.35	1.36	1.33	1.26	1.28	1.32	2.5	0.122	0.37	5
cis-1,3-Dichloropropene	1.32	1.34	0.97	1.20	1.18	1.18	1.12	1.09	1.18	2.5	0.120	0.36	5
4-Methyl-2-pentanone	5.87	5.29	3.12	4.56	4.50	4.42	4.29	4.59	4.58	6.3	0.794	2.38	13

Reporting Limit = Low Level Standard

NOTE: Attachment is subject to change without notice.
NOTE: The estimated detection limit is 1/5 the reporting limit.

Attachment 3 (continued)

CompuChem Method Detection Limit Study

Study date: March 25, 2004		GCMS Volatile SW846 8260B/5030B/5035 Soil, 5 gm Purge with Sodium Bisulfate												
Instrument: 5972hp52														
Compound Name	Rep#1	Rep#2	Rep#3	Rep#4	Rep#5	Rep#6	Rep#7	Rep#8	Mean	Test Conc.	SDev	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
Toluene	1.39	1.60	1.29	1.55	1.53	1.45	1.37	1.35	1.44	2.5	0.109	0.33	5
trans-1,3-Dichloropropene	1.27	1.27	0.91	1.12	1.08	1.09	1.06	1.06	1.11	2.5	0.118	0.35	5
1,1,2-Trichloroethane	1.81	1.73	1.34	1.75	1.71	1.76	1.75	1.82	1.71	2.5	0.154	0.46	5
Ethylmethacrylate	71.97	72.02	69.53	71.57	71.38	71.15	70.62	71.01	71.16	25	0.809	2.43	50
1,3-Dichloropropane	1.44	1.48	1.12	1.52	1.40	1.41	1.30	1.39	1.38	2.5	0.125	0.37	5
Tetrachloroethene	1.32	1.43	1.22	1.51	1.42	1.40	1.31	1.28	1.36	2.5	0.095	0.28	5
2-Hexanone	12.13	11.07	8.96	10.00	9.98	10.18	9.75	10.37	10.31	6.3	0.945	2.83	13
Dibromochloromethane	1.42	1.48	1.12	1.40	1.38	1.34	1.27	1.34	1.34	2.5	0.110	0.33	5
1,2-Dibromoethane	1.54	1.56	1.17	1.47	1.53	1.48	1.41	1.50	1.46	2.5	0.125	0.38	5
Chlorobenzene	1.34	1.44	1.22	1.46	1.43	1.42	1.34	1.32	1.37	2.0	0.081	0.24	5
1,1,1,2-Tetrachloroethane	1.31	1.30	1.08	1.40	1.33	1.28	1.25	1.31	1.28	2.5	0.093	0.28	5
Ethylbenzene	1.13	1.24	1.02	1.19	1.20	1.16	1.08	1.14	1.15	2.5	0.070	0.21	5
m,p-Xylene	2.33	2.60	2.18	2.68	2.54	2.55	2.34	2.30	2.44	4.0	0.175	0.52	10
o-Xylene	1.18	1.27	1.03	1.25	1.23	1.18	1.10	1.16	1.18	2.5	0.080	0.24	5
Styrene	2.34	2.32	2.16	2.31	2.28	2.25	2.25	2.18	2.26	2.5	0.065	0.19	5
Bromoform	1.58	1.53	1.15	1.52	1.52	1.52	1.42	1.49	1.47	2.5	0.135	0.41	5
Isopropyl benzene	1.14	1.20	1.01	1.23	1.18	1.18	1.08	1.05	1.13	2.5	0.079	0.24	5
1,1,2,2-Tetrachloroethane	4.65	4.31	3.98	4.33	4.34	4.31	4.28	4.32	4.32	2.5	0.180	0.54	5
1,2,3-Trichloropropane	2.05	1.82	1.25	1.90	1.85	1.88	1.73	1.75	1.78	2.0	0.235	0.71	5
Bromobenzene	1.31	1.41	1.16	1.41	1.40	1.38	1.30	1.29	1.33	2.5	0.086	0.26	5
trans-1,4-Dichloro-2-butene	20.72	20.73	19.91	20.60	20.52	20.51	20.32	20.45	20.47	10	0.264	0.79	20
n-Propyl benzene	0.95	1.04	0.91	1.15	1.08	0.97	0.91	0.95	1.00	2.5	0.087	0.26	5
2-Chlorotoluene	1.29	1.35	1.06	1.35	1.31	1.26	1.28	1.20	1.26	2.5	0.095	0.29	5
4-Chlorotoluene	1.12	1.15	1.00	1.14	1.13	1.06	1.00	0.96	1.07	2.5	0.075	0.22	5
1,3,5-Trimethyl benzene	1.06	1.10	0.89	1.11	1.08	1.05	1.00	0.96	1.03	2.5	0.076	0.23	5
tert-butyl Benzene	1.59	1.64	1.32	1.64	1.65	1.57	1.50	1.45	1.55	2.5	0.116	0.35	5
1,2,4-Trimethyl benzene	2.17	2.20	2.01	2.22	2.21	2.19	2.10	2.10	2.15	2.5	0.073	0.22	5
sec-butyl Benzene	1.02	1.19	0.98	1.12	1.22	1.08	1.02	0.97	1.08	2.5	0.094	0.28	5
1,3-Dichlorobenzene	1.06	1.20	0.95	1.18	1.10	1.09	1.06	1.00	1.08	2.5	0.084	0.25	5
p-Isopropyl toluene	0.96	1.01	0.80	0.99	1.03	0.94	0.90	0.83	0.93	2.5	0.083	0.25	5
1,4-Dichlorobenzene	1.43	1.43	1.20	1.48	1.55	1.48	1.40	1.42	1.42	2.5	0.102	0.31	5
1,2-Dichlorobenzene	1.31	1.32	1.08	1.40	1.38	1.34	1.30	1.27	1.30	2.5	0.096	0.30	5
n-Butyl benzene	0.95	1.01	0.77	0.94	1.01	0.92	0.88	0.84	0.92	2.5	0.083	0.25	5
1,2-Dibromo-3-chloropropane	1.80	1.84	1.25	1.85	1.93	1.86	1.76	1.91	1.78	2.5	0.219	0.66	5
1,2,4-trichlorobenzene	1.20	1.17	0.93	1.14	1.22	1.15	1.10	1.04	1.12	2.0	0.095	0.28	5
Hexachlorobutadiene	1.17	1.12	0.95	1.14	1.20	1.12	1.03	1.03	1.10	2.0	0.084	0.25	5
Naphthalene	1.53	1.56	1.28	1.63	1.69	1.63	1.52	1.56	1.55	2.0	0.124	0.37	5
1,2,3-Trichlorobenzene	1.47	1.43	1.17	1.42	1.50	1.39	1.31	1.33	1.38	2.0	0.106	0.32	5

Reporting Limit = Low Level Standard

NOTE: Attachment is subject to change without notice.
NOTE: The estimated detection limit is 1/5 the reporting limit.

Attachment 4

COMPUCHEM a division of Liberty Analytical Corp DATE ___/___/___INITIAL TIME OF TUNE_____ SHIFT/S(A)_____(B)_____(C)_____
GC/MS VOLATILE RUN LOG TIME TUNE EXPIRES_____ LINKER /METHOD_____
COMPUCHEM LOGBOOK 11 X 17 (F50052)
PREVENTIVE MAINTENANCE_____

	REFLECTED	FILE NAME	REPORTED	DATE	TIME	CLIENT ID#	CASE/SDG#	SAMPLE VOLUME	CHEMIST	COMMENTS(ETC.)/DISPOSITION
1				/ /						
2				/ /						
3				/ /						
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23				/ /						
24				/ /						

SUPERVISOR APPROVAL_____ Date_____

Tune (ID #7008) Lot No. _____

Calibration Group Code / Lot No. _____

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/30/02:dce

Note: Attachment is subject to change without notice.

Attachment 5

TCL4 182 HIGH

RESTEK

CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30431 Lot No.: A027506
Description: 502.2 CAL 2000 Mega-Mix
Expiration Date[1]: August 2005 Storage: Freezer

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1,1-dichloroethane	75-34-3	99%	2,000 µg/ml	+/- .04%
1,1-dichloroethene	75-35-4	99%	2,000 µg/ml	+/- .04%
1,1-dichloropropene	563-58-6	99%	2,000 µg/ml	+/- .04%
1,1,1-trichloroethane	71-55-6	99%	2,000 µg/ml	+/- .04%
1,1,1,2-tetrachloroethane	630-20-6	99%	2,000 µg/ml	+/- .04%
1,1,2-trichloroethane	79-00-5	99%	2,000 µg/ml	+/- .04%
1,1,2,2-tetrachloroethane	79-34-5	99%	2,000 µg/ml	+/- .04%
1,2-dibromo-3-chloropropane	96-12-8	97%	2,000 µg/ml	+/- .04%
1,2-dibromoethane	106-93-4	99%	2,000 µg/ml	+/- .04%
1,2-dichlorobenzene	95-50-1	99%	2,000 µg/ml	+/- .04%
1,2-dichloroethane	107-06-2	99%	2,000 µg/ml	+/- .04%
1,2-dichloropropane	78-87-5	99%	2,000 µg/ml	+/- .04%
1,2,3-trichlorobenzene	87-61-6	99%	2,000 µg/ml	+/- .04%
1,2,3-trichloropropane	96-18-4	99%	2,000 µg/ml	+/- .04%
1,2,4-trichlorobenzene	120-82-1	99%	2,000 µg/ml	+/- .04%
1,2,4-trimethylbenzene	95-63-6	99%	2,000 µg/ml	+/- .04%
1,3-dichlorobenzene	541-73-1	99%	2,000 µg/ml	+/- .04%
1,3-dichloropropane	142-28-9	99%	2,000 µg/ml	+/- .04%
1,3,5-trimethylbenzene	108-67-8	99%	2,000 µg/ml	+/- .04%
1,4-dichlorobenzene	106-46-7	99%	2,000 µg/ml	+/- .04%
2-chlorotoluene	95-49-8	99%	2,000 µg/ml	+/- .04%
2,2-dichloropropane	594-20-7	99%	2,000 µg/ml	+/- .04%
4-chlorotoluene	106-43-4	99%	2,000 µg/ml	+/- .04%
p-isopropyltoluene	99-87-6	99%	2,000 µg/ml	+/- .04%
benzene	71-43-2	99%	2,000 µg/ml	+/- .04%
bromobenzene	108-86-1	99%	2,000 µg/ml	+/- .04%
bromochloromethane	74-97-5	99%	2,000 µg/ml	+/- .04%

[1]Expiration date of the unopened ampul stored at recommended temperature.

[2]Listed in alphabetical order.

[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.

[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).

[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS (cont.)

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

PAGE 2

Cat. No.: 30431 Lot No.: A027506

Description: 502.2 CAL 2000 Mega-Mix

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
bromodichloromethane	75-27-4	99%	2,000 µg/ml	+/- .04%
bromoform	75-25-2	99%	2,000 µg/ml	+/- .04%
carbon tetrachloride	56-23-5	99%	2,000 µg/ml	+/- .04%
chlorobenzene	108-90-7	99%	2,000 µg/ml	+/- .04%
chloroform	67-66-3	99%	2,000 µg/ml	+/- .04%
cis-1,2-dichloroethene	156-59-2	99%	2,000 µg/ml	+/- .04%
cis-1,3-dichloropropene	10061-01-5	99%	2,000 µg/ml	+/- .04%
dibromochloromethane	124-48-1	99%	2,000 µg/ml	+/- .04%
dibromomethane	74-95-3	99%	2,000 µg/ml	+/- .04%
ethylbenzene	100-41-4	99%	2,000 µg/ml	+/- .04%
hexachlorobutadiene	87-68-3	99%	2,000 µg/ml	+/- .04%
isopropylbenzene	98-82-8	99%	2,000 µg/ml	+/- .04%
methylene chloride	75-09-2	99%	2,000 µg/ml	+/- .04%
m-xylene	108-38-3	99%	2,000 µg/ml	+/- .04%
naphthalene	91-20-3	99%	2,000 µg/ml	+/- .04%
n-butylbenzene	104-51-8	99%	2,000 µg/ml	+/- .04%
n-propylbenzene	103-65-1	99%	2,000 µg/ml	+/- .04%
o-xylene	95-47-6	99%	2,000 µg/ml	+/- .04%
p-xylene	106-42-3	99%	2,000 µg/ml	+/- .04%
sec-butylbenzene	135-98-8	99%	2,000 µg/ml	+/- .04%
styrene	100-42-5	99%	2,000 µg/ml	+/- .04%
tetrachloroethene	127-18-4	99%	2,000 µg/ml	+/- .04%
tert-butylbenzene	98-06-6	99%	2,000 µg/ml	+/- .04%
toluene	108-88-3	99%	2,000 µg/ml	+/- .04%
trans-1,2-dichloroethene	156-60-5	99%	2,000 µg/ml	+/- .04%
trans-1,3-dichloropropene	10061-02-6	97%	2,000 µg/ml	+/- .04%
trichloroethene	79-01-6	99%	2,000 µg/ml	+/- .04%
Solvent: purge & trap methanol	67-56-1	99%		

[1]Expiration date of the unopened ampul stored at recommended temperature.

[2]Listed in alphabetical order.

[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.

[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

TCL4 - GASES HIGH

RESTEK

CERTIFICATE OF ANALYSIS

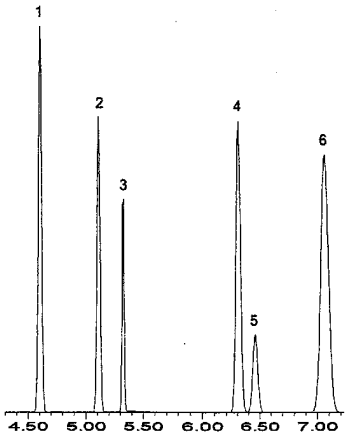
110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30042 Lot No.: A028050
Description: 502.2 Calibration Mix #1
Expiration Date[1]: March 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	Dichlorodifluoromethane	75-71-8	99%	1,997 µg/ml	± 1.9%
2	Chloromethane	74-87-3	99%	2,003 µg/ml	± 1.3%
3	Vinyl chloride	75-01-4	99%	2,003 µg/ml	± 1.4%
4	Bromomethane	74-83-9	99%	1,997 µg/ml	± 1.3%
5	Chloroethane	75-00-3	99%	2,003 µg/ml	± 1.5%
6	Trichlorofluoromethane	75-69-4	99%	2,000 µg/ml	± 0.3%
Solvent:	Purge and trap Methanol	67-56-1	99%		

Column: 105m .32mm 1.8µm **Rtx-502.2 (cat.#10921)**
Carrier gas: helium @ 2.2 ml/min.
Temp. program: 50°C (hold 12 min.) Isothermal
Inj. temp.: 240°C
Det. temp.: 240°C
Detector type: MSD



John Lidgett
John Lidgett—QA Analyst

[1]*Expiration date of the unopened ampul stored at recommended temperature.*
[2]*Listed in alphabetical order.*
[3]*Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.*
[4]*Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*
[5]*Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.*

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

TCL4- KETONES HIGH

AccuStandard Inc.

125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9863-R1
DESCRIPTION: Custom VOA Std #2
LOT #: B3110072
SOLVENT: Methanol

EXPIRATION: Feb 9, 2004

See reverse for additional certification information.

This product is guaranteed accurate to + 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetone	67-64-1	99.9	1261 ±50.44	1260
Methyl ethyl ketone	78-93-3	100	1261 ±50.44	1261
4-methyl-2-pentanone	108-10-1	99.4	1261 ±50.44	1253
2-hexanone	591-78-6	100	1261 ±50.44	1261
Vinyl acetate	108-05-4	99.9	500.8 ±20.03	500.3
2-Chloroethylvinyl ether	110-75-8	99.7	500.3 ±20.01	498.8
Acrolein	107-02-8	90.0	5575 * ±223.00	5018
Acrylonitrile	107-13-1	99.9	5000 ±200.00	4995

8 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty u(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is U(y) *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00
Rev. 11/0

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

TCL4-APPIX H1

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9862-R1
DESCRIPTION: Custom VOA Std #1
LOT #: B3110080
SOLVENT: Methanol

EXPIRATION: Feb 27, 2004

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetonitrile	75-05-8	99.9	500.3 ±20.01	499.8
Allyl chloride	107-05-1	99.6	500.0 ±20.00	498.0
1-Chlorohexane	544-10-5	99.0	500.3 ±20.01	495.3
Chloroprene	126-99-8	98	500.0 ±20.00	490.0
trans-1,4-Dichloro-2-butene	110-57-6	97.4	25,670 *±1026.80	25003
p-Dioxane	123-91-1	99.2	25,010 ±1000.40	24810
Ethyl methacrylate	97-63-2	99.0	5010 ±200.40	4960
Isobutyl alcohol	78-83-1	100	25,010 ±1000.40	25010
Methacrylonitrile	126-98-7	99.9	5010 ±200.40	5005
Methyl iodide	74-88-4	100	500.3 ±20.01	500.3
Methyl methacrylate	80-62-6	99.8	5003 ±200.12	4993
Pentachloroethane	76-01-7	97.3	514.0 *±20.56	500.1
Propionitrile	107-12-0	99.9	25,010 ±1000.40	24985

13 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

RESTEK

CERTIFICATE OF ANALYSIS

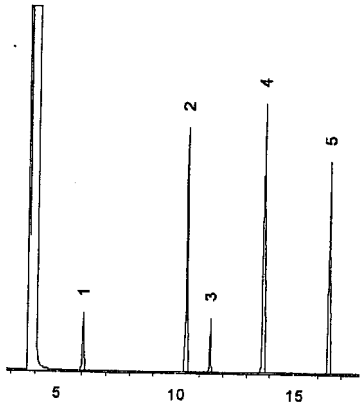
110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY—READ MSDS PRIOR TO USE.

Cat. No.: 30005 Lot No.: A019743
Description: VOA Matrix Spike Mix
Expiration Date[1]: January 2005

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	1,1-dichloroethene	75-35-4	99%	2,500 µg/ml	± 0.2%
2	benzene	71-43-2	99%	2,500 µg/ml	± 0.2%
3	trichloroethene	79-01-6	99%	2,500 µg/ml	± 0.2%
4	toluene	108-88-3	99%	2,500 µg/ml	± 0.2%
5	chlorobenzene	108-90-7	99%	2,500 µg/ml	± 0.2%
Solvent:	purge and trap methanol	67-56-1	99%		

Column: 105m .53mm 3.0µm Rtx®-502.2 (cat.#10910)
Carrier gas: hydrogen @ 40cm/sec
Temp. program: 40°C (hold 2 min.) to 240°C @ 8°C/min. (hold 10 min.)
Inj. temp.: 200°C
Det. temp.: 250°C
Detector type: FID



John Lidgett
John Lidgett—QA Analyst

[1]Expiration date of the unopened ampul stored at recommended temperature.
[2]Listed in alphabetical order.
[3]As determined by capillary GC/FID unless otherwise noted. Value rounded to the nearest LOWER whole percentage. In addition to GC/FID, chemical identity and purity are confirmed using 2 or more of the following: GC/MS, solid probe MS, GC/ECD, GC/FPD, GC/NPD, GC/TC, HPLC, DSC, FTIR, melting point, refractive index, and Karl Fisher.
[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 Issued by DNV Certification, Inc.

NOTE: Attachment is subject to change without notice.

Attachment 6

Laboratory Name/North Carolina Certificate Number: CompuChem/79															
Analyst: Joann Ockerlander															
Study Date: January 14, 2003															
Method: 8260B Soil 5 gm purge															
Instrument/Column/Detector: 5972hp59															
Compounds	TrueVal	Rep #1	Rep #2	Rep #3	Rep #4	Mean	Mean	SOP	SD(n-1)	-3SD	+3SD	-3SD	+3SD	RSD	SOP
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	% R	%R	ug/l	of (x)	of (x)	% R	%R	%	%RSD
Dichlorodifluoromethane	50	50.93	37.94	35.22	32.64	39.18	78	60-140	8.13	14.8	64	38	162	21	30
Chloromethane	50	51.27	42.97	43.09	39.77	44.28	89	21-150	4.91	29.5	59	67	133	11	15
Vinyl Chloride	50	56.50	45.37	48.16	42.10	48.03	96	43-138	6.16	29.5	67	61	139	13	15
Bromomethane	50	61.95	51.71	56.04	51.13	55.21	110	48-150	5.00	40.2	70	73	127	9	15
Chloroethane	50	56.89	49.18	54.30	44.44	51.21	102	53-144	5.53	34.6	68	68	132	11	15
Trichlorofluoromethane	50	52.25	47.39	47.09	45.24	47.99	96	60-140	2.99	39.0	57	81	119	6	15
1,1-Dichloroethene	50	58.45	51.06	52.90	49.79	53.05	106	72-129	3.82	41.6	65	78	122	7	15
1,1,1-trichloro-2,2,2-trichloroethane	50	56.46	48.70	53.04	51.12	52.33	105	80-120	3.28	42.5	62	81	119	6	15
Acetone	125	149.5	116.9	118.3	104.0	122.2	98	39-150	19.35	64.1	180	52	148	16	15
Iodomethane	50	47.73	41.27	51.66	38.88	44.89	90	80-120	5.87	27.3	62	61	139	13	15
1,1,2-trichloro-1,2,2-trichloroethane	50	54.90	48.56	52.48	48.89	51.21	102	80-120	3.04	42.1	60	82	118	6	15
Carbon disulfide	50	56.75	50.21	51.52	47.25	51.43	103	20-150	3.97	39.5	63	77	123	8	15
3-Chloropropene	50	56.70	51.43	49.95	44.67	50.69	101	80-120	4.95	35.8	66	71	129	10	15
Acetonitrile	50	54.05	52.27	48.53	44.11	49.74	99	80-120	4.40	36.5	63	73	127	9	15
Methyl acetate	50	56.34	46.53	47.13	42.27	48.07	96	80-120	5.92	30.3	66	63	137	12	15
Methylene Chloride	50	54.25	52.31	51.67	48.25	51.62	103	78-136	2.50	44.1	59	85	115	5	15
Acrylonitrile	500	611.1	523.8	495.0	447.4	519.3	104	80-120	68.82	312.8	726	60	140	13	15
trans-1,2-Dichloroethene	50	55.84	51.80	52.35	51.47	52.86	106	74-125	2.02	46.8	59	89	111	4	15
Methyl-tert-butyl ether	50	54.61	49.28	48.12	46.09	49.52	99	64-145	3.63	38.6	60	78	122	7	15
1,1-Dichloroethane	50	54.69	50.75	50.61	50.00	51.51	103	71-130	2.14	45.1	58	88	112	4	15
Chloroprene	50	47.41	50.92	45.50	44.42	47.06	94	80-120	2.86	38.5	56	82	118	6	15
Vinyl acetate	50	54.97	50.41	49.60	44.13	49.78	100	60-140	4.45	36.4	63	73	127	9	15
Isopropyl ether	50	53.81	51.45	51.10	46.89	50.82	102	80-120	2.88	42.2	59	83	117	6	15
cis-1,2-Dichloroethene	50	52.18	51.90	51.52	50.05	51.41	103	74-134	0.95	48.6	54	94	106	2	15
2,2-Dichloropropane	50	53.25	48.23	49.93	48.11	49.88	100	80-120	2.40	42.7	57	86	114	5	15
Propionitrile	2500	3002	2469	2453	2347	2568	103	80-120	294.7	1684	3452	66	134	11	15
2-butanone	125	155.03	126.44	123.35	113.08	129.47	104	48-150	17.97	75.6	183	58	142	14	15
Methacrylonitrile	250	283.2	256.5	239.0	222.4	250.3	100	80-120	26.00	172.3	328	69	131	10	15
Bromochloromethane	50	55.93	51.87	51.21	51.44	52.61	105	60-140	2.23	45.9	59	87	113	4	15
Chloroform	50	51.35	50.87	49.95	48.32	50.12	100	70-131	1.33	46.1	54	92	108	3	15
1,1,1-Trichloroethane	50	53.45	48.66	50.23	49.07	50.35	101	70-134	2.17	43.9	57	87	113	4	15
Cyclohexane	50	53.23	46.02	51.86	47.39	49.62	99	80-120	3.47	39.2	60	79	121	7	15
1,1-dichloropropene	50	55.57	49.31	50.93	49.77	51.39	103	80-120	2.86	42.8	60	83	117	6	15
Carbon Tetrachloride	50	53.35	47.33	49.76	49.79	50.06	100	72-137	2.48	42.6	58	85	115	5	15
1,2-Dichloroethane	50	50.03	47.75	46.43	45.98	47.55	95	63-139	1.82	42.1	53	89	111	4	15
Isobutyl alcohol	2500	3207	2299	2304	2266	2519	101	60-140	458.8	1142	3895	45	155	18	30
Benzene	50	53.88	51.00	51.69	49.64	51.55	103	75-125	1.77	46.2	57	90	110	3	15
Trichloroethene	50	57.94	50.76	52.51	53.35	53.64	107	66-147	3.06	44.5	63	83	117	6	15
Methylcyclohexane	50	51.45	43.79	46.01	46.61	46.97	94	80-120	3.23	37.3	57	79	121	7	15
1,2-Dichloropropane	50	53.03	49.86	50.74	47.96	50.40	101	71-130	2.10	44.1	57	87	113	4	15
Dibromomethane	50	55.19	53.23	52.60	51.19	53.05	106	60-140	1.66	48.1	58	91	109	3	15
1,4-dioxane	2500	3162	2425	2453	2587	2657	106	60-140	344.5	1623	3690	61	139	13	15
Methylmethacrylate	500	558.1	496.4	476.8	470.3	500.4	100	80-120	40.05	380.3	621	76	124	8	15
Bromodichloromethane	50	52.09	50.15	48.73	48.78	49.94	100	68-135	1.58	45.2	55	91	109	3	15
cis-1,3-Dichloropropene	50	53.24	50.28	50.92	50.17	51.15	102	73-139	1.43	46.9	55	92	108	3	15
4-Methyl-2-pentanone	125	147.9	126.1	122.4	102.6	124.7	100	51-133	18.58	69.0	180	55	145	15	15
Toluene	50	53.58	49.80	50.99	46.11	50.12	100	78-126	3.11	40.8	59	81	119	6	15
trans-1,3-Dichloropropene	50	50.08	49.16	48.89	43.73	47.96	96	64-144	2.87	39.4	57	82	118	6	15
1,1,2-Trichloroethane	50	54.05	50.54	48.86	45.49	49.73	99	73-127	3.56	39.1	60	79	121	7	15
Ethylmethacrylate	500	535.8	498.4	474.4	436.0	486.2	97	80-120	41.89	360.5	612	74	126	9	15
1,3-Dichloropropane	50	53.87	49.56	49.49	43.78	49.18	98	80-120	4.14	36.8	62	75	125	8	15
Tetrachloroethene	50	56.50	47.87	53.46	48.50	51.58	103	79-131	4.12	39.2	64	76	124	8	15
2-hexanone	125	148.7	120.4	119.8	102.2	122.8	98	53-133	19.25	65.0	181	53	147	16	30
Dibromochloromethane	50	52.31	49.19	48.81	45.84	49.04	98	82-127	2.64	41.1	57	84	116	5	15
1,2-Dibromoethane	50	55.69	48.80	49.44	46.49	50.11	100	80-120	3.93	38.3	62	76	124	8	15
Chlorobenzene	50	53.33	48.85	50.90	48.22	50.33	101	80-117	2.31	43.4	57	86	114	5	15
1,1,1,2-Tetrachloroethene	50	53.65	49.62	51.56	47.39	50.56	101	80-120	2.68	42.5	59	84	116	5	15
Ethylbenzene	50	52.26	48.44	50.16	47.32	49.55	99	82-117	2.16	43.1	56	87	113	4	15
m,p-Xylene	100	101.0	94.12	97.93	94.16	96.79	97	81-115	3.30	86.9	107	90	110	3	15
o-Xylene	50	51.32	47.39	50.74	47.99	49.36	99	74-120	1.96	43.5	55	88	112	4	15
Styrene	50	52.02	48.98	49.14	47.40	49.39	99	77-119	1.92	43.6	55	88	112	4	15
Bromoform	50	55.58	49.13	50.40	47.11	50.56	101	65-1.39	3.62	39.7	61	79	121	7	15
Isopropyl Benzene	50	48.95	45.00	46.10	44.83	46.22	92	80-120	1.91	40.5	52	88	112	4	15
1,1,2,2-Tetrachloroethene	50	52.22	46.71	44.65	43.88	46.87	94	55-141	3.76	35.6	58	76	124	8	15
1,2,3-Trichloropropane	50	55.64	45.47	46.60	44.37	48.02	96	80-120	5.16	32.5	63	68	132	11	15

NOTE: Attachment is subject to change without notice.

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) _____

- Procedure Code: IP 480B SOP Section #: 1.3.2.2 Revision #: 11

SOP Title: Analysis of Volatile Organic Compounds in Aqueous and Medium / High Concentration Soil Samples by SW-846

Effective date: (QA fills in) 3/27/06

- Procedure prepared by: [signature] Date: 3/21/06
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 3-23-06
- Reason for change: Addition of DoD-QSM Requirements
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition Update III, Method 8260B, Method 5030B and Method 5035B; New York State Analytical Services Protocol (NYSASP), June 2000, Plus Revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

_____ _____

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 480B: Analysis of Volatile Organic Compounds in Aqueous and Medium/High Concentration Soil Samples by SW-846

Table of Contents

Instrument Procedure 480B: Analysis of Volatile Organic Compounds in Aqueous and Medium/High Concentration Soil Samples by SW-846

1.0 Scope and Application

This is a general purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in a variety of aqueous matrices following SW-846 Method 8260B. It is also used to analyze medium to high level soil samples that have been extracted with methanol. The method is applicable to a wide range of organic compounds. It incorporates Method 5030B and Method 5035 (for medium/high concentration soil samples, extracted with methanol). Target compounds for this method, along with their associated internal standards and quantitation ions, are listed in Attachment 1. Note, however, that many of these compounds are not routinely analyzed.

The reporting limit is the low level calibration standard concentration. **Reporting limits for this method are shown in Attachment 2.**

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

An inert gas (helium) is bubbled through a 5 mL or 25 mL aqueous sample spiked with internal standard and surrogate compounds. For medium/high level soil samples, an aliquot **(typically 100 µL)** of the methanol extract containing surrogates **(See SOP 1.1.4.1)** is added to 5 mL nitrogen-sparged reagent water. The sample is purged in a **40 mL VOA vial** at ambient temperature, causing the purgeable volatile organic compounds to be transferred from the aqueous phase to the vapor phase. The vapor is swept onto a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and back-flushed with the inert gas to desorb the purgeables onto a gas chromatograph (GC) wide-bore capillary column. The GC is temperature-programmed to separate the purgeables that are then detected with a mass spectrometer (MS).

Note: For Method 8260B, a heated purge is also allowed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements.

3.3 Reporting Units – **µ**g/L for water and **µ**g/**Kg** for soil

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: **The DoD-QSM and South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for the DoD-QSM and 10% for the SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.**

3.5 SC DHEC – South Carolina Department of Health and Environmental Control

3.6 DoD-QSM – Department of Defense Quality Systems Manual

3.7 Marginal Exceedance – Beyond the LCS control limit but within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

4.0 Interferences

4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, it may be necessary to reduce solvent contamination in the laboratory, purge the methanol used to prepare standard solutions, purge the DI water with helium or nitrogen, change the purge gas source, or regenerate the molecular sieve purge gas filter.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples are stored in GC/MS VOA laboratory refrigerator**s**; separate from laboratory standards, and they must be analyzed in a room in which the atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. Because methylene chloride will permeate PTFE tubing, all GC carrier gas lines and purge gas plumbing are to be constructed from stainless steel or copper tubing.

4.3 Contamination by carryover can occur whenever a sample is analyzed after a sample that contains high levels of organic compounds. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank or the next sample must be closely monitored to check for cross-contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high purgeable levels, it may be necessary to clean the purge and trap apparatus. Do so by purging a 10-20% methanol solution, followed by baking the purge and trap apparatus and the analysis of a DI water blank to confirm that the system is free from contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bake out and purging of the entire system may be required.

4.4 Instrument Problems/Preventative Maintenance

Instrument problems may interfere with the analysis. If a low response is observed for the early eluting compounds such as the gases, replacement of the trap or septum may be necessary. In addition, adjustments to the purge flow may be necessary to achieve a desired response for these compounds. If such adjustments do not help, it may be necessary to check the fittings on the purge and trap device and on the column for leaks. This is done with a helium leak detector and certain software utility programs. Column maintenance or replacement may be necessary if peak tailing or broad chromatographic peaks are observed.

5.0 Safety

5.1 The toxicity and carcinogenicity of many chemicals used in this method have not been precisely determined. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be minimized. Preparation of calibration standards, blanks, and samples is performed in a fume hood to minimize risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2-dichloroethane, 1,1,2,2-tetrachloroethane, 1,1,2- trichloroethane, chloroform, 1,2-dibromoethane, trichloroethene, and vinyl chloride.

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Syringes

6.1.1 1 mL Hamilton syringe

6.1.2 500 **µL** Hamilton syringe

6.1.3 100 **µL** Hamilton syringe

6.1.4 50 **µL** Hamilton syringe

6.1.5 25 **µL** Hamilton syringe

6.2 Volumetric Flasks and Pipets

6.2.1 Assorted volumetric flasks ranging from 50 mL to 1000 mL

6.2.2 10 mL graduated pipette in 1/10 mL graduations

6.2.3 One **mL** mininert vials

6.3 Analytical Column

6.3.1 Supelco SPB-624 75 m, 0.32 mm ID with 1.8 µm film thickness

6.3.2 Restek RTX-VMS 20 m, 0.18 mm ID with 1 µm film thickness

6.4 **GC**/MS

6.4.1 The MS scans 35-300 amu at 0.7 second scan time in the electron impact mode at 70 eV (nominal).

6.4.2 Finnigan INCOS 500 mass spectrometers

6.4.3 Hewlett Packard 5890 GC

6.4.4 Hewlett Packard 6890 GC

6.4.5 Hewlett Packard 5972 **and 5973 Mass Selective Detectors (MSD)**

6.4.6 Varian 3400 GC

6.5 Interface (GC to MS)

6.5.1 Type:

6.5.1.1 Jet separator **(Finnigan INCOS 500)**

6.5.1.2 Direct capillary interface **(HP 5972 and 5973 MSD)**

6.5.2 Temperature: 250° C

6.6 Data System

6.6.1 A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.6.2 The data processing software searches any GC/MS data file for ions of a specified mass and plots abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software integrates the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, the software compares sample spectra against reference library spectra. The reference library used is the NIST Mass Spectral Library.

6.6.3 For data acquisition, the INCOS 500 systems use Prolab acquisition software on Pentium computers.

6.6.4 For data acquisition, the Hewlett Packard systems use ChemStation software on Pentium computers.

6.6.5 For data processing, the Hewlett Packard HP 9000 series 735 Unix Workstation employing Target3 and Envision software by Thru-Put Systems is used.

6.6.6 Data is stored on the Target3 processing system on a short-term basis. Data is stored on a log-term basis on data servers.

6.9 Purge and Trap

6.9.1. Archon Model 5100, 4552 Purge and trap autosampler **interfaced** directly to a Tekmar 3000 Purge and Trap Concentrator

6.9.**2** **Supelco K (Carbopak B, Carboxen 1000 and 1001)**

7.0 Reagents and Standards

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methanol (B&J Scientific, purge and trap grade)

7.3 Tuning Standard

7.3.1 4-Bromofluorobenzene - Standard ID# 7008 at 25 µg/**mL**. **2 µL** yielding 50 **η**g on column, are injected onto the column every 12 hours.

7.3.1.1 Prepare the standard by adding 50 **µL** Restek VOA Tuning Mix 5000 µg/m**L**) to an amount of purge and trap grade methanol in a 10 **mL** volumetric flask and bring to volume.

7.4.1.2 Prepare this standard monthly.

7.4 Calibration Standards

7.4.1 For the initial calibration, the internal standard solution is added automatically by the Archon Purge and Trap Autosampler. For all subsequent analyses, both the internal standard and the surrogate solutions are added automatically by the Archon autosampler.

Standard Preparation

Std. ID TCL4-High	**005 µg/L**	**010 µg/L**	**020 µg/L**	**050* µg/L**	**100 µg/L**	**200 µg/L**
8260 I.S.	5.0 **µL**	5.0 **µL**	5.0 **µL**	5.0 **µL**	5.0 **µL**	5.0 **µL**
8260 S.S.	1.0 **µL**	2.0 **µL**	5.0 **µL**	10.0 **µL**	15.0 **µL**	20.0 **µL**
TCL4-1&2	1.0 **µL**	2.0 **µL**	4.0 **µL**	10.0 **µL**	20.0 **µL**	40.0 **µL**
TCL4-gases	1.0 **µL**	2.0 **µL**	4.0 **µL**	10.0 **µL**	20.0 **µL**	40.0 **µL**
TCL4-ketones	1.0 **µL**	2.0 **µL**	4.0 **µL**	10.0 **µL**	20.0 **µL**	40.0 **µL**
TCL4-AppIX	1.0 **µL**	2.0 **µL**	4.0 **µL**	10.0 **µL**	20.0 **µL**	40.0 **µL**

* Continuing Calibration level

7.4.1.1 To prepare the standards at the concentrations shown in the column headers of **preceding table** , add the **µL** amount of standard shown

to a 100 **mL** volumetric flask containing nitrogen sparged DI water, then bring up to volume.

Alternatively, the standards may be prepared at the above concentrations by diluting the 200 µg/L standard directly into the purge and trap impingers. Dilute as follows:

- For a 100 µg/L standard, add 2.5 **mL** to 5 **mL** DI water;
- For a 50 µg/L standard, add 1.25 **mL** to 5 **mL** DI water;
- For a 20 µg/L standard, add 0.50 **mL** to 5 **mL** DI water;
- For a 10 µg/L standard, add 0.25 **mL** to 5 **mL** DI water;
- For a 5 µg/L standard, add 0.125 **mL** to 5 **mL** DI water.

For a 25 **mL** purge analysis, increase both volumes by a factor of 5 when preparing standards by making dilutions.

Note: For samples submitted to meet the regulatory requirements of the State of South Carolina, this initial calibration curve must include a standard at 2 µg/L.

7.4.1.2 The concentration of the compounds in the TCL4-1 & 2 High standard is 500 µg/**mL**. See Attachment 5 for the composition of this standard.

7.4.1.2.1 Prepare the standard by adding 1.25**mL** Restek 502.2 VOA 2000 MegaMix (2000 µg/**mL**) to an amount of purge and trap grade methanol in a 5 **mL** volumetric flask and bring to volume.

7.4.1.2.2 Prepare this standard every three months.

7.4.1.3 The concentration of the compounds in the TCL4-gases High standard is 500 µg/**mL**. See Attachment 5 for the composition of this standard.

7.4.1.3.1 Prepare each standard by adding 1.25 mL Restek 502.2 Calibration Mix #1 (2000 µg/**mL**) to an amount of purge and trap grade methanol in a 5 **mL** volumetric flask and bring to volume.

7.4.1.3.2 The method states that this standard usually needs to be replaced weekly, unless the standard manufacturer recommends otherwise, or unless the acceptability of the

standard can be documented. This standard has generally proven to be more stable in the laboratory. The gas standard can be used for longer than a week if the gases in the continuing calibration (CCV) standard meet the CCV requirements when compared to the initial calibration standards that contain a gas standard that has been prepared within a one week holding time. Prepare this standard monthly, or more frequently as need dictates, or when degradation is evident rendering the standard unacceptable.

7.4.1.4 The concentration of the compounds in the TCL4-ketones standard is 500 µg/mL, 1250 µg/mL, or 5000 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.4.1 Prepare the standard by transferring AccuStandard custom VOA Mix #2 to a mininert vial.

7.4.1.4.2 Replenish this standard as needed and replace every three months.

7.4.1.5 The concentration of the compounds in the TCL4-AppIX standard is 500 µg/mL, 5000 µg/mL, or 25000 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.5.1 Prepare the standard by transferring AccuStandard custom VOA Mix #1 to a mininert vial.

7.4.1.5.2 Replenish this standard as needed and replace every three months.

7.5 Standard Storage

7.5.1 Store the stock standards in Teflon- sealed screw-cap bottles with zero headspace at -10° C to -20° C. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases should be monitored and fresh standards prepared if a 20% drift is experienced. These standards need to be replaced after six months or as recommended by the manufacturer, unless the acceptability of the standard can be documented. CEVE and styrene may have to be prepared more frequently.

7.5.2 Store secondary dilution standards in Teflon®-sealed screw-cap bottles with minimal headspace at -10° C to -20° C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards.

7.5.3 Aqueous working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device. If premixed certified solutions are used store according to manufacturer's documented holding time and storage temperature recommendations.

7.5.4 Purgeable standards are stored in GC/MS VOA Freezer #1, separate from other standards and samples.

8.0 Sample Preservation **and** Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.1.1 Note that if 2-chloroethyl vinyl ether **(CEVE)** is a target compound of interest for the project, an unpreserved sample must be analyzed within 7 days of collection.

8.2 All samples must be analyzed within 14 days of collection.

8.3 Prior to analysis, all samples must be stored under refrigeration at 2 - 4^{o} C in the reach-in storage unit in the laboratory. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 All samples, QC, and standards are spiked with the surrogate standard. Surrogates are used to assess the efficiency of the analytical system.

9.1.2 Surrogate compounds must meet the following percent recovery criteria.

Surrogate Compound	Aqueous and Med./High Conc. Soil	Aqueous

	5 mL purge	25 mL purge
Dibromofluorobenzene	71-141	65-150
1,2-**Dichloroethane-d_4**	70-139	59-150
Toluene-**d_8**	72-123	61-145
4-bromofluorobenzene	65-131	63-143

9.1.2.1 The same surrogates and recovery criteria are to be used for samples submitted to meet the regulatory requirements of the State of South Carolina.

9.1.2.2 The following table contains the surrogate recovery limits required by the DoD-QSM.

Surrogate Compound	Aqueous	Solid
Dibromofluorobenzene	**85-115**	**N/A**
1,2-Dichloroethane-d_4	**70-120**	**N/A**
Toluene-d_8	**85-120**	**85-115**
4-Bromofluorobenzene	**75-120**	**85-120**

9.2 Internal Standards

9.2.1 The integrated areas of the quantitation ions of the internal standards are monitored in continuing calibration verification checks, samples, and QC for a change in retention time and response or sensitivity. These should remain reasonably constant over time.

9.2.1.1 Internal standard retention time and area responses must be assessed in each continuing calibration verification standard by comparison to the corresponding internal standard in the most recent initial calibration mid-point standard. Internal standard responses in samples and QC are compared to the most recent continuing calibration verification.

9.2.2 The area responses of the internal standards must be within 50 - 200% difference of the area responses compared to.

9.2.3 The retention time shift for the internal standards must be less than 30 seconds.

9.2.4 If any of these criteria cannot be met, the analytical system must be checked for malfunctions and corrections made. Re-analysis of any affected sample is required.

9.3 Method/Instrument Blanks

9.3.1 Before any samples are analyzed, it must be demonstrated through a laboratory reagent blank that the system is free of contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbents, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next analysis. In general, background **contamination** from method analytes should be below the reporting limit.

9.3.2 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration verification acceptance criteria.

9.3.3 A method blank is analyzed with each batch of up to 20 samples processed as a group within a 12-hour tune. If more than 20 samples are analyzed in a tune batch, a second method blank is required. Method blanks must be analyzed immediately following a valid continuing calibration verification analysis. For SC DHEC a blank is required every 10 samples.

9.3.**4** The concentration of the target compounds in the blank must be less than the reporting limit for each target compound.

9.3.4.1 The DoD-QSM requires that the target compounds in the blank must be at a concentration of <½ the reporting limit and < the reporting limit for lab contaminants.

9.3.4.2 The SC DHEC requires that all compounds in the blank be less that the reporting limits, except common lab contaminates can be twice the reporting limit.

9.3.**5** All samples processed within the same 12-hour tune with a method blank that does not meet the blank technical acceptance criteria must be reanalyzed. The chromatographic system must be inspected for malfunctions, and corrections must be made as required before more samples are analyzed.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS) is prepared and analyzed with each tune batch of up to 20 samples. The LCS and matrix spike**s** are spiked with the same target analytes. The LCS is spiked at the same concentration as the matrix spike. For SC DHEC the LCS is analyzed every 10 samples. **The LCS is spiked with all compounds of interest in the project.**

9.4.2 The percent recovery criteria, developed from in-house statistical data, for the remainder of the analytes in the full LCS are listed in Attachment 3.

9.4.2.1 The LCS control limits required by the DoD-QSM are listed in Attachment 6 along with the marginal exceedances.

9.4.2.2 The following table presents the DoD-QSM and 2003 NELAC Standards allowed number of marginal exceedances governed by the number of compounds spiked into the LCS.

Number of analytes in the LCS	Allowed number of Marginal Exceedances
> 90	**5**
71-90	**4**
51-70	**3**
31-50	**2**
11-30	**1**
< 11	**0**

The LCS fails if the more than the allowed number of marginal exceedances occur or if a spike recovery is outside of the marginal exceedance limits.

9.4.**3**. Gases and known poor purging compounds

- gases: bromomethane
  chloromethane
  chloroethane
  vinyl chloride
  dichlorodifluoromethane
  trichlorofluoromethane

- acetone
- 2-butanone
- carbon disulfide

- crotonaldehyde
- 1,2-dibromo-3-chloropropane
- 1,4-dioxane
- isobutyl alcohol
- 2-hexanone
- 4-methyl-2-pentanone
- vinyl acetate

9.4.**4** For SC DHEC, an expanded subset of analytes, representative of the compounds being reported, is employed and all analytes must have recovery limits within 70-130%. The analytes are:

- vinyl chloride
- 1,1-dichloroethene
- methylene chloride
- 1,1-dichloroethane
- cis-1,2-dichloroethene
- 2-butanone
- carbon tetrachloride
- benzene
- trichloroethene
- 1,2-dichloropropane
- bromodichloromethane
- tetrachloroethene
- chlorobenzene
- ethylbenzene
- styrene
- bromoform
- 1,4-dichlorobenzene

9.4.**5** When the LCS fails to meet the acceptance criteria, the entire batch associated with it must be re-prepared and reanalyzed.

9.5 Matrix Spikes

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared and analyzed with every SDG. For SC DHEC the MS/MSD must be analyzed every 10 aqueous samples.

9.5.2 Matrix spikes have the following advisory recovery criteria as shown in **the following table**.

Spike Compound	Aqueous & med./high conc. soil % Recovery
1,1-dichloroethene	61-145
Trichloroethene	71-120
Benzene	76-127
Toluene	76-125
Chlorobenzene	75-130
All others	50-150

9.5.3 Matrix spikes have the following advisory relative percent difference (RPD) criteria as shown in **the following table**.

Spike Compound	Aqueous & med./high conc. soil % RPD
1,1-dichloroethene	14
Trichloroethene	14
Benzene	11
Toluene	13
Chlorobenzene	13
All others	25

9.5.4 Most spike compounds should meet these criteria. If the criteria are not met in the MS/MSD but are met in the LCS, the results may be reported with the failures attributed to the matrix of the sample. If the LCS does not meet criteria, then all will have to be repeated as discussed above.

9.5.5 To meet the requirements of the DoD-QSM, the duplicate matrix spikes should meet the LCS control limits listed in Attachment 6. The RPD between the duplicate matrix spike should be ≤ 30%.

9.5.5.1 If the duplicate matrix spikes fail DoD-QSM acceptance criteria, contact the client for guidance.

9.5.5.2 If original sample results are associated with failing duplicate matrix spikes, qualify the results in the narrative as estimated concentrations. Refer to the DoD-QSM "J" flag.

9.6 Duplicates

9.6.1 Duplicates, at a frequency of 10%, are required when processing samples submitted to meet the regulatory requirements of SC DHEC. This can be satisfied with the MS/MSD.

9.7 Initial Calibration Verification

9.7.1 A second source initial calibration verification (ICV) standard is run after the initial calibration standards have met criteria.

9.7.2 The ICV must be within 20% of its expected value for each target analyte and surrogate or within 40% for the poor purgers and the gases. Sporadic failure of up to three target compounds is allowed but they must not exceed 40% of their expected value; gases and poor purgers are listed above.

9.7.2.1 To meet the requirements for the DoD-QSM, the ICV must be ≤ 25% of its expected value for each target analyte.

9.7.3 If the ICV fails to meet the criteria in 9.7.2 or 9.7.2.1, take corrective action and reanalyze the standard. If the ICV fails again, repeat the initial calibration.

9.8 MDL **Studies**

9.8.1 On an annual basis **and after major maintenance,** a method detection limit (MDL) study is performed on a**t least one** instrument per method and matrix. When multiple instruments are used, individual instrument MDL studies may **be** replaced by the analysis of an MDL check sample. The MDL check sample must be analyzed on all instruments, to demonstrate equivalent sensitivity.

9.8.1.1 The DoD-QSM requires that the MDL check sample is prepared at about 2x the MDL and is analyzed on a quarterly basis for each matrix. A response must be detected in the 2x MDL check sample. Additionally, qualifying ions of 50% or higher must also be present. For more information on MDL studies, refer to QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies."

9.9 Contingency

9.9.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.9.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.9.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.9.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 BFB Tuning

10.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2 **µL** STD ID#7008) into the GC using a 10 **µL** Hamilton syringe. BFB may be analyzed simultaneously with a continuing calibration verification standard as long as all QC criteria are met.

10.1.2 The peak selection criteria for BFB analysis are as follows (in order of performance):

10.1.2.1 Average one scan prior to the apex of the BFB peak to one scan after the apex, subtracting a single background scan prior to the peak, but no more than 20 scans prior to the elution of BFB. Also, do not subtract part of the BFB peak.

10.1.2.2 Choose the apex of the BFB peak only and include background subtraction.

Note: Background subtraction is performed to eliminate interference and when performed, the subtracted scan must be no more than 20 scans prior to the elution of the

BFB and no scans within the BFB peak may be subtracted.

10.1.2.3 Choose a single scan or a range of scans within the BFB peak and include background subtraction.

10.1.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in **the following table**.

BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15-40% of m/z 95
75	30-60% of m/z 95
95	Base Peak; 100% relative abundance
96	5-9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5-9% of m/z 174
176	>95% but less than 101% of m/z 174
177	5-9% of m/z 176

10.1.**4** BFB technical acceptance criteria must be met before any standards, samples, or required blanks are analyzed. GC/MS tuning and Mass Calibration forms must be printed and attached to the instrument run log page for each tune. The relative abundance for each ion is calculated to two decimal places.

10.1.**5** If BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective action to achieve the technical acceptance criteria.

10.2 Initial Calibration

10.2.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution (BFB) criteria have been met, each GC/MS system must be calibrated at six concentrations to demonstrate

instrument sensitivity and the linearity of responses for the purgeable target compounds.

10.2.2 Prepare standards according to the Initial Calibration Standard Preparation Table 2 in Section 7.4. All initial calibration standards must be analyzed at the concentration levels and frequency described in this SOP on a GC/MS system **that** meets the BFB technical acceptance criteria.

10.2.3 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard. Relative response factors (RRF) are calculated for each compound.

10.2.**4** Minimum relative response factors for the System Performance Check Compounds (SPCC) must be met and are listed **in the following table**.

Relative Response Factor Criteria for SPCCs

Volatile Compound	**Minimum RRF**
Chloromethane	0.10
1,1-dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

10.2.**5** **The** following **Calibration Check Compounds (CCC)** have maximum % RSD criteria of ≤ 30%.

- 1,1-dichloroethene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene
- vinyl chloride

10.2.**5**.1 **The remaining compounds must have an RSD of ≤ 15%.** If the % RSD is 15% or less, the average relative response factor may be used for quantitation. If the % RSD is greater than 15% then an alternate method for quantitation, such as a linear calibration using least squares regression or quadratic fit, may

be used. When one of these options is used, the line must not be forced through the origin**.**

If the linear or quadratic regression fit is used, the correlation coefficient must be ≥ 0.990. These alternate methods of quantitation are available in the ThruPut system.

Note: The DoD-QSM requires that the linear least squares regression correlation coefficient must be ≥ 0.995 and does not allow the used of quadratic fit.

10.2.**5**.2 **If the initial calibration does not meet the criteria above, corrective action is necessary.**

- Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the column, service the purge and trap device, or take other corrective action to achieve the technical acceptance criteria.

- Compare responses for the analyte in each of the standard levels to verify that a single standard analysis is not **presenting results significantly higher or lower then the other standard analyses, as this would indicate that the standard solution was prepared in error. If that is the case, re-prepare and reanalyze the standard.**

10.2.**6** The initial calibration may still be acceptable when some analytes exceed the 15% RSD criteria, if the following conditions are met and allowed by the client:

- The mean of <u>all</u> %RSD values for the analytes (grand mean) is less than or equal to 15%.

- <u>All</u> analytes in the calibration standard must be included in the calculation.

- Non-CCC target compounds have a warning limit of 50% RSD and an action limit of 90% RSD when the "grand mean" approach is used. These limits have been inserted as default values into the data reduction software program. This is based strictly on established U.S.

EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.2.**6**.1 A summary of the initial calibration data and/or a list of the analytes not meeting the 15% RSD criteria with their actual %RSD must be included as a deliverable to our client. If the conditions in 10.2.7 are met, then the average relative response factor may be used to determine the concentration of analytes in samples.

Note: For samples submitted to meet the regulatory requirements of **both** the State of South Carolina **and the DoD-QSM,** the grand mean option is not allowed**.**

10.2.**7** The initial calibration verification must be analyzed after each initial calibration and must meet the acceptance criteria. If the ICV fails, then a new initial calibration curve must be generated.

10.3 Continuing Calibration Verification

10.3.1 Before the analysis of samples and blanks, but after BFB and initial calibration acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration verification standard. This standard must contain all purgeable target analytes and surrogate compounds. It is used to ensure that the instrument meets the sensitivity and linearity requirements of the method throughout the analytical sequence.

10.3.2 A check of the calibration curve must be performed once every 12 hours, beginning with the injection of BFB. A percent difference of the response for each compound compared to the mean relative response factor from the initial calibration is calculated when performing the average response factor model.

10.3.3 The calculated percent difference must be less than or equal to 20% (%D) for the CCCs listed above in Section 10.2.**5**. Minimum response factor criteria for the continuing calibration verification standard are also shown **in Section 10.2.4**.

10.3.3.1 If a regression fit model was used for analytes in the initial calibration, the continuing calibration verification is performed using percent drift (difference) for the CCCs.

10.3.4 As indicated for the initial calibration acceptance criteria, for the continuing calibration verification, the remaining target analytes (non-CCC compounds) do not have defined % difference criteria. We have established a warning limit of 50%D and an action limit of 90%D. These values have been inserted as defaults into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.3.4.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, the non-CCC target analytes should be less than 50%.

10.3.5 If continuing calibration verification acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration will have to be performed.

Note: Method 8260B indicates that if the CCCs are not required analytes, then all required analytes, must meet the 20% difference criterion. Our typical analysis includes all of the CCCs.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. All injections must be recorded on the instrument run log (Attachment 4) along with the date, time (use a 24 hour clock), the volume injected, operator ID, and any comments relevant to the injection.

All standards, blanks, samples and other required runs must be injected within exactly 12 hours from the time of the injection of BFB.

11.1 Instrument Software Conventions

11.1.1 Quantitation method: Average **response factor**

11.1.2 File naming convention**s**:

11.1.2.1 Name calibration standards and instrument blanks as follows:

XXYYMMDDZZ

where: XX = **BF, BG… for BFB file names**

XX = CB, CC… for instrument blank file names

XX = CS, CT… for calibration standards

YY = two digit year

MM = two digit month

DD = two digit day

Y = **shift (A, B, or C indicating 1st, 2nd, or 3rd shift)**

ZZ = **two digit i**nstrument number

11.1.2.2 Name sample analyses as follows:

CCN##R# or CCN##D#

where: CCN = work order number designated for the sample by LIMs

= the sample number (typically 01-20)

R# = indicating reanalysis of the sample (R1, R2…)

D# = indicating diluted analysis of the sample (D1, D2…)

11.2 Analytical Sequence

11.2.1 Order of analysis for the instrument calibration

- BFB (tune)
- initial calibration
- initial calibration verification

11.2.2 Order of analysis for the twelve-hour tune

- BFB
- continuing calibration verification
- instrument blank
- laboratory control sample
- samples

11.2.3 In some cases, if tune time remains after the initial calibration standards have been run, samples may be analyzed as long as they are preceded by a**n acceptable** instrument blank **and LCS**.

11.2.4 All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.3 Preparations

11.3.1 Standards

11.3.1.1 Load the standard solutions onto the purge and trap apparatus. Purge for 11 minutes at ambient temperature, or 40° C for heated purge, and desorb for 4 minutes, analyzing all target compounds.

11.3.2 Instrument Blank

11.3.2.1 An instrument blank is prepared by filling one 25 **mL** or 5 mL gastight syringe with DI water and spiking with internal standard solution and surrogate solution.

11.3.3 Laboratory Control Sample spikes

11.3.3.1 Laboratory control samples (LCS) are prepared at 5.0 µg/L with 5 µg/L of surrogates for 25 mL samples and at 50 µg/L with 50 µg/L of surrogates for 5 mL samples.

11.3.3.2 For certain projects and programs, a full list spike is required.

11.3.4 Samples

11.3.4.1 Liquid samples are collected with zero headspace and provided to the laboratory in 40 mL or 60 mL screw-cap vials with Teflon-lined silicon septa. To prepare liquid samples for analysis, pour the sample into a 5 mL or 25 mL Hamilton gastight syringe. Replace the plunger and adjust the volume to 5 mL or 25 mL. Spike each 25 mL sample with 2.5 **µL** of internal standard and 2.5 **µL** of surrogate solutions through the bore of the syringe. Spike each 5 mL sample with 5 **µL** of internal standard and 5 **µL** of surrogate solutions. **Inject syringe contents into a 40 mL vial and place on the autosampler for analysis.**

11.3.4.2 Methanol extracts of medium/high concentration soil samples are contained in an autosampler vial capped with a Teflon-faced septum. The vial contains 1 mL of the methanol extract that also contains surrogates. A 5 mL Hamilton gastight syringe is filled with DI water and adjusted to 4.9 mL. The plunger is pulled back to 5.0 mL to allow for the addition of the methanol extract and internal standard solution. Add 100 **µL** of the methanol extract and 5.0 **µL** of internal standard solution to the syringe. Based on prior or screening results, if less than 100 **µL** of the methanol extract is required in order to get target analytes within the initial calibration range, add an additional amount of methanol to total 100 **µL**. **Inject the contents of the syringe into a 40 mL vial and place on the autosampler for analysis.**

11.3.4.3 For further details, see Sample Preparation Procedure –238, "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap."

11.3.6 Matrix Spikes

11.3.6.1 For **matrix** spikes, in addition to spiking internal standard solution and surrogate solution, also add 5.0 **µL** of 8260B spike solution. For certain projects, a full target list matrix spikes are required. For medium/high concentration soil samples, the spiking solution is added at the time of the methanol extraction.

11.4 Analysis

11.4.**1** Purge the sample for 11.0 (± 0.1) minutes.

11.4.**2** After purging, the purge and trap apparatus will desorb onto the GC column by elevating the trap temperature to 260° C and back-flushing the trap with helium for 4 minutes at 20 to 60 mL/minute.

11.4.**3** After desorbing, the trap is reconditioned by baking at 260° C for at least 7 minutes. When the trap has finished baking and is cool, it is ready for the next sample to be purged.

11.4.**4** In each analytical run, all analytes must fall below the maximum calibration range established by the highest standard in the initial calibration. If an analyte is present at a concentration higher than the

highest initial calibration standard, it must be reanalyzed at a lesser amount or dilution. A valid dilution is one in which the compound in question falls **within** the mid **and high** point calibration standard concentration.

11.5 Identification

11.5.1 Target compounds are identified in the samples by analyzing standards under the same condition. Resultant mass spectra are compared to established library spectra and GC retention times are compared to retention times from the most recent continuing calibration standard. The mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria.

11.5.1.1 All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.

11.5.1.2 The relative intensities of ions specified above must agree within ± 20% between the library and sample spectra.

11.5.1.3 Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for.

11.5.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory will report that identification.

11.5.3 Non-target compounds (tentatively identified compounds or TICs) are identified by comparing the mass spectra from the TICs to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library.

11.6 Quantitation

11.6.1 A relative response factor is established for each target and surrogate compound during the initial and continuing calibration verification procedures. Quantitation of target analytes is based on the mean relative response factor from the initial calibration curve.

11.6.**2** Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative.

11.6.**3** TICs are quantified by comparing the mass spectral response from the reconstructed ion chromatogram (RIC) for the TIC peaks to the mass spectral response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis and Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ \mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} x\ 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax\ x\ C(is)}{A(is)\ x\ Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured
A(is) = Area of the characteristic ion (EICP) for the specific internal standard
C(is) = Concentration of the internal standard (in µg/l)
Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.9 Concentration

12.9.1 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard.

12.9.2 Concentration of aqueous samples by GC/MS analysis using relative response factor:

$$\mu g / L = \frac{(Ax)(Is)(Df)}{(Ais)(\overline{RRF})(Vo)}$$

where: Ax = area of the characteristic ion from the EICP for the compound to be measured
Ais = area of the characteristic ion for the EICP for the internal standard
Is = amount of internal standard added (ng)
$\overline{RRF}$ = mean relative response factor from initial calibration standards
Vo = volume of water purged in milliliters
Df = dilution factor. If no dilution, Df = 1.0

12.9.3 Concentration of medium level soil samples (dry weight basis) by GC/MS

$$\mu g / kg = \frac{(Ax)(Is)(Df)(Vt)(1000)}{(Ais)(\overline{RRF})(Ws)(Va)(D)}$$

where: Ax, Ais, Is, $\overline{RRF}$ are the same as given for water

Df = Dilution factor which, for medium/high concentration soil extract is defined by the following formula:

$$\frac{\mu L \text{ concentrated extract used for dilution} + \mu L \text{ clean solvent}}{\mu L \text{ concentrated extract used for dilution}}$$

Vt = Total volume of methanol extract, in mL

Note: This is typically 10 mL or 5 mL even though only 1 mL is transferred to the autosampler vial.

Ws = Weight of soil/sediment sample extracted, in grams (g)
Va = Volume of the aliquot of methanol extract

Note: Typically this is 100 **µL** but can be the volume of sample extract (not including the methanol added to equal 100 **µL**) in **µL** added to DI water.

Ws = weight of sample extracted, in grams

$$D \text{ (dry weight)} = \frac{100 - \% \text{ moisture}}{100}$$

12.9.4 Concentration of aqueous and soil samples by GC/MS using quadratic (second order) fit in Target:

$$y = [n][b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)]$$

where: b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
x = area of analyte/area of Internal Standard
n = amount of Internal Standard
y = concentration in ng on column
Rsp = area of analyte/area of Internal Standard

Example: Area of acetone = 35659
Area of IS = 613275
b = -0.0909161
m^1 = 9.605304
m^2 = 7.132688
ηg of IS = 250
response = 35659/613275 = 0.058145

Amount in **η**g on column =

$$(250\ \eta g)[-0.0909161 + 9.605304\ x\ 0.058145 + 7.132688\ x\ 0.058145\ \text{^}2] = 122.9\eta g$$

Concentration (water) $\mu g / L = \dfrac{(122.9\eta g)(Df)}{(Vo)}$

Concentration (soil) $\mu g / Kg = \dfrac{122.9\eta g}{(Ws)(D)}$

12.9.5 Concentration of aqueous and soil samples by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\dfrac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where: As = Area of the target analyte peak in the sample

Ais = Area of the internal standard peak
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

$$\text{Concentration (water) } \mu g / L = \frac{(Cs)(Df)}{(Vo)}$$

$$\text{Concentration (soil) } \mu g / Kg = \frac{Cs}{(Ws)(D)}$$

12.9.6 Tentatively Identified Compound (TIC) Estimation

$$TIC\ Amount\ (water)\ \mu\mathbf{g/L} = \frac{(Area\,TIC)\ \mathbf{x}\ Amount\ (Std)(Df)}{Area(IS)\ x\ 1(RF)\ \mathbf{x}\ (Vo)}$$

$$TIC\ Amount\ (soil)\ \mu\mathbf{g/Kg} = \frac{(Area\,TIC)\ \mathbf{x}\ Amount\ (Std)(Df)(Vt)}{Area\ (IS)\ x\ 1(RF)\ \mathbf{x}\ (Vi)(Ws)(D)}$$

where:
- Area (TIC) = area response from RIC for non-target compound
- Amount (Std) = amount of internal standard added to the sample, in μg/L.
- Area (IS) = area response of the nearest internal standard in the reconstructed ion chromatogram
- 1(RF) = assumed response factor of 1

12.10 Calculating Dilutions

12.10.1 The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters of water purged (i.e., Vo above) to the number of milliliters of the original water sample used for purging. For example, if 12.5 mL of sample is diluted to 25.0 mL with DI water and purged, Df = 25.0 mL/12.5 mL = 2.0. If no dilution is performed, Df = 1.0.

If a methanol extract contains an analyte that exceeds the high level standard a dilution must be performed. Determine a level of dilution

that will result in a value within the upper half of the calibration range. This is an acceptable dilution. Example: A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 8260B, Methods 5030B and 5035

16.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.**3** Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.**4** QCSOP: Proper Documentation Procedures

16.**5** QCSOP: Numerical Data Reduction

16.**6** "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.**7** Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.**8** NELAC Standards, **June 2003**, plus revisions

16.**9** QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006

16.**11** New York State Environmental Laboratory Approval Program, Certification Manual, **April 2005**, plus revisions.

16.**12** CompuChem Quality Manual, Revision **7**, **Update 1, 12/13/05**, plus revisions

16.**13** Sample Control SOP 4.1, "Receiving Samples"

16.**14** Sample Control SOP 4.6, "Storing Samples"

16.**15** QC SOP 13.11, Performing Annual Method Detection Limit (MDL) Studies

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Target Analyte List

17.2 Attachment 2 **– Target Analyte Reporting** Limits

17.3 Attachment 3 – **In-house** Statistical Control Limits

17.4 Attachment 4 – Instrument Run Log

17.5 Attachment 5 – Standard Certificate of Analysis

17.6 Attachment 6 – **DoD-QSM LCS Control Limits**

Attachment 1

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
dichlorodifluoromethane	1	85	87
chloromethane	1	50	52
vinyl chloride	1	62	64
bromomethane	1	94	96
chloroethane	1	64	66
trichlorofluoromethane	1	101	103
diethyl ether	1	74	45, 59
1,1-dichloroethene	1	96	61, 98
methylene chloride	1	84	49, 86
trans-1,2-dichloroethene	1	96	61, 98
1,1-dichloroethane	1	63	65, 83
2,2-dichloropropane	1	77	97
cis-1,2-dichloroethene	1	96	61, 98
bromochloromethane	1	128	49, 130
chloroform	1	83	85, 47
1,1,1-trichloroethane	1	97	99, 61
carbon tetrachloride	1	117	119, 121
1,1-dichloropropene	1	75	110, 77
benzene	1	78	77, 51
1,2-dichloroethane	1	62	98, 64
trichloroethene	1	130	95, 97
1,2-dichloropropane	1	63	39, 41
dibromomethane	1	174	93, 95
bromodichloromethane	1	83	85, 127
2-chloroethyl vinyl ether	1	63	65, 106
cis-1,3-dichloropropene	1	75	77, 110
acrolein	1	56	55, 38
iodomethane	1	142	127, 141
1,1,1-trichloro-2,2,2,-trifluoroethane	1	117	151, 153
1,1,2-trichloro-1,2,2,-trifluoroethane	1	85	101, 151
carbon disulfide	1	44	78, 76
acetone	1	43	58
3-chloropropene	1	39	41, 76
acetonitrile	1	41	39, 38
acrylonitrile	1	53	52, 51

Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
methyl-tert-butyl ether	1	73	41, 43
vinyl acetate	1	43	86
2-butanone	1	43	57, 72
propionitrile	1	54	55, 52
methacrylonitrile	1	41	39, 67
1,4-dioxane	1	88	58
methylmethacrylate	1	69	100, 41
Surrogate #1: dibromofluoromethane	1	113	111, 192
Surrogate #2: 1,2-dichloroethane-$\mathbf{d_4}$	1	65	102, 67
4-methyl-2-pentanone	2	43	85, 100
toluene	2	92	91
trans-1,3-dichloropropene	2	75	77, 110
1,1,2-trichloroethane	2	97	83, 85
ethylmethacrylate	2	69	41, 99
tetrachloroethene	2	164	168, 129
1,3-dichloropropane	2	76	78
2-hexanone	2	43	58, 71
dibromochloromethane	2	129	127, 48
1,2-dibromoethane	2	107	109, 81
chlorobenzene	2	112	114, 77
1,1,1,2-tetrachloroethane	2	131	119, 133
ethylbenzene	2	106	91
m,p-xylene	2	106	91
o-xylene	2	106	91
styrene	2	104	78, 103
bromoform	2	173	175, 254
isopropyl benzene	2	105	120
bromobenzene	2	156	77,158
1,1,2,2-tetrachloroethane	2	83	85, 131
1,2,3-trichloropropane	2	110	75, 112
trans-1,4-dichloro-2-butene	2	53	88, 90
Surrogate #3: d_8-toluene	2	98	70, 100

Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
n-propyl benzene	3	91	120
2-chlorotoluene	3	126	91
4-chlorotoluene	3	126	91
1,2,4-trimethyl benzene	3	105	120
1,3,5-trimethyl benzene	3	105	120
Pentachloroethane	3	167	130, 165
sec-butyl benzene	3	105	134
1,2-dichlorobenzene	3	146	111, 148
1,3-dichlorobenzene	3	146	111, 148
1,4-dichlorobenzene	3	146	111, 148
n-butyl benzene	3	91	92, 134
tert-butyl benzene	3	119	91, 134
p-isopropyl toluene	3	119	134, 91
1,2-dibromo-3-chloropropane	3	157	75, 39
1,2,4-trichlorobenzene	3	180	182, 145
hexachlorobutadiene	3	225	223, 227
naphthalene	3	128	64, 51
1,2,3-trichlorobenzene	3	180	182, 145
Surrogate #4: 4-bromofluorobenzene	3	95	174, 176
Internal Standard #1: fluorobenzene	NA	96	70
Internal Standard #2: chlorobenzene-$\mathbf{d_5}$	NA	117	82, 119
Internal Standard #3: 1,4-dichlorobenzene-$\mathbf{d_4}$	NA	152	150

Attachment 2

Compound Name	Reporting Limits Med Level Soil/5 mL Aqueos Purge µg/Kg & µg/L
Dichlorodifluoromethane	5
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
Acrolein	50
1,1-Dichloroethene	5
1,1,1-Trichloro-2,2,2-trifluoroetha	5
Acetone	13
Iodomethane	5
1,1,2-trichloro-1,2,2-trifluoroethan	5
Carbon disulfide	5
3-Chloropropene	5
Acetonitrile	5
Methyl acetate	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2-Dichloroethene	5
Methyl-tert-butyl-ether	5
1,1-Dichloroethane	5
Chloroprene	5
Vinyl acetate	5
Isopropyl ether	5
cis-1,2-Dichloroethene	5
2,2'-Dichloropropane	5
Propionitrile	250
2-Butanone	13
Methacrylonitrile	5
Bromochloromethane	5
Chloroform	5
1,1,1-Trichloroethane	5
Cyclohexane	5
1,1-Dichloropropene	5
Carbon tetrachloride	5
1,2-Dichloroethane	5
Isobutyl alcohol	250
Benzene	5
Trichloroethene	5
Methylcyclohexane	5
1,2-Dichloropropane	5
Dibromomethane	5
1,4-Dioxane	250
Methylmethacrylate	50
Bromodichloromethane	5
2-Chloroethyl vinyl ether	5

Attachment 2 (continued)

Compound Name	Reporting Limits Med Level Soil/5 mL Aqueos Purge µg/Kg & µg/L
cis-1,3-Dichloropropene	5
4-Methyl-2-pentanone	13
Toluene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Ethylmethacrylate	5
1,3-Dichloropropane	5
Tetrachloroethene	5
2-Hexanone	13
Dibromochloromethane	5
1,2-Dibromoethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5
m,p-Xylene	10
o-Xylene	5
Styrene	5
Bromoform	5
Isopropyl benzene	5
1,1,2,2-Tetrachloroethane	5
1,2,3-Trichloropropane	5
Bromobenzene	5
trans-1,4-Dichloro-2-butene	100
n-Propyl benzene	5
2-Chlorotoluene	5
4-Chlorotoluene	5
1,3,5-Trimethyl benzene	5
tert-butyl Benzene	5
1,2,4-Trimethyl benzene	5
sec-butyl Benzene	5
1,3-Dichlorobenzene	5
p-Isopropyl toluene	5
1,4-Dichlorobenzene	5
1,2-Dichlorobenzene	5
n-Butyl benzene	5
1,2-Dibromo-3-chloropropane	5
1,2,4-trichlorobenzene	5
Hexachlorobutadiene	5
Naphthalene	5
1,2,3-Trichlorobenzene	5
1,2-Dichloroethene (total)	10
Xylene (total)	15

Attachment 2 (continued)

Reporting Limits	
Compound Name	**8260B 25 mL Purge µg/L**
Dichlorodifluoromethane	0.5
Chloromethane	0.5
Vinyl Chloride	0.5
Bromomethane	0.5
Chloroethane	0.5
Trichlorofluoromethane	0.5
Diethyl ether	5.0
Acrolein	5.0
1,1-Dichloroethene	0.5
1,1,1-trichloro-2,2,2-trifluoroethane	0.5
1,1,2-trichloro-1,2,2-trifluoroethane	0.5
Acetone	2.5
Iodomethane	0.5
Carbon disulfide	0.5
3-Chloropropene	0.5
Acetonitrile	0.5
Methyl acetate	0.5
Methylene chloride	0.5
Acrylonitrile	5.0
trans-1,2-Dichloroethene	5.0
Methyl-tert-butyl-ether	0.5
n-hexane	0.5
1,1-Dichloroethane	0.5
Vinyl acetate	1.0
Isopropyl ether	0.5
2,2'-Dichloropropane	0.5
cis-1,2-Dichloroethene	0.5
Chloroprene	0.5
2-Butanone	2.5
Propionitrile	25
Methyl acrylate	5.0
Methacrylonitrile	5.0
Bromochloromethane	0.5
Chloroform	0.5
Tetrahydrofuran	2.0
1,1,1-Trichloroethane	0.5
Pentafluorobenzene	0.5
Cyclohexane	0.5
1-Chlorobutane	0.5
Isobutyl alcohol	25
1,1-Dichloropropene	0.5
Carbon tetrachloride	0.5
Benzene	0.5
1,2-Dichloroethane	0.5
Crotonaldehyde	5.0
Trichloroethene	0.5
Methylcyclohexane	0.5
1,2-Dichloropropane	0.5
Dibromomethane	0.5
Methylmethacrylate	5.0
1,4-Dioxane	25
Bromodichloromethane	0.5
2-chloroethyl vinyl ether	5.0
cis-1,3-Dichloropropene	0.5

Attachment 2 (continued)

Reporting Limits	
Compound Name	**8260B 25 mL Purge µg/L**
4-Methyl-2-pentanone	2.5
Toluene	0.5
trans-1,3-Dichloropropene	0.5
Ethylmethacrylate	5.0
1,1,2-Trichloroethane	0.5
1,3-Dichloropropane	0.5
Tetrachloroethene	0.5
2-Hexanone	2.5
Dibromochloromethane	0.5
1,2-Dibromoethane	0.5
1-Chlorohexane	0.5
Chlorobenzene	0.5
1,1,1,2-Tetrachloroethane	0.5
Ethylbenzene	0.5
m,p-Xylene	0.5
o-Xylene	0.5
Styrene	0.5
Bromoform	0.5
Isopropyl benzene	0.5
cis-1,4-dichloro-2-butene	20
1,1,2,2-Tetrachloroethane	0.5
Bromobenzene	0.5
1,2,3-Trichloropropane	0.5
trans-1,4-dichloro-2-butene	20
n-Propyl benzene	0.5
2-Chlorotoluene	0.5
1,3,5-Trimethylbenzene	0.5
4-Chlorotoluene	0.5
tert-butyl benzene	0.5
Pentachloroethane	0.5
1,2,4-Trimethylbenzene	0.5
sec-Butyl Benzene	0.5
1,3-Dichlorobenzene	0.5
p-Isopropyl toluene	0.5
1,4-Dichlorobenzene	0.5
Benzyl chloride	0.5
n-Butyl benzene	0.5
1,2-Dichlorobenzene	0.5
1,2-Diethylbenzene	0.5
1,2-Dibromo-3-chloropropane	0.5
1,2,4-trichlorobenzene	0.5
Hexachlorobutadiene	0.5
Naphthalene	0.5
1,2,3-Trichlorobenzene	0.5
1,2-Dichloroethene (total)	0.5
Xylene (total)	1.0

Attachment 3

Statistical Control Limits for the LCS

Compound	Percent Recovery Range	
	25 mL purge	**5 mL purge & med./high conc. Soil**
Dichlorodifluoromethane	50-150	50-150
Chloromethane	50-150	51-150
Vinyl chloride [2]	61-150	54-136
Bromomethane	50-150	59-150
Chloroethane	54-150	55-150
Trichlorofluoromethane	56-150	55-150
1,1-dichloroethene [1, 2]	74-143	70-130
Carbon disulfide	50-150	66-146
1,1,2-trichloro-1,2,2,-fluoroethane	78-150	59-138
Methylene chloride [2]	50-139	72-123
trans-1,2-dichloroethene	50-137	39-130
Methyl-tert-butyl ether	68-134	62-135
1,1-dichloroethane [2]	59-138	70-126
cis-1,2-dichloroethene [2]	69-140	70-131
2-butanone [2]	65-134	64-127
Chloroform	67-147	65-133
1,1,1-trichloroethane	71-137	69-134
Carbon tetrachloride [2]	68-145	70-139
Benzene [1, 2]	68-138	69-130
1,2-dichloroethane	61-150	67-133
Trichloroethene [1, 2]	55-150	72-130
1,2-dichloropropane [2]	67-137	73-123
Bromodichloromethane [2]	73-142	71-126
cis-1,3-dichloropropene	74-134	63-136
4-methyl-2-pentanone	66-127	51-141
Toluene [1]	60-142	61-131
trans-1,3-dichloropropene	66-130	60-140
1,1,2-trichloroethane	68-130	63-131
Tetrachloroethene [2]	65-137	78-136
2-hexanone	53-140	50-143
Dibromochloromethane	68-137	70-129
1,2-dibromoethane	73-128	61-137
Chlorobenzene [1, 2]	68-129	76-121

Attachment 3 (continued)

Statistical Control Limits for the LCS

Compound	Percent Recovery Range	
	25 mL purge	**5 mL purge & med./high conc. Soil**
Ethylbenzene [2]	**67-127**	**68-131**
Styrene [2]	**66-139**	**73-127**
Bromoform [2]	**62-139**	**66-133**
Isopropyl benzene	56-143	62-138
1,1,2,2-tetrachloroethane	63-122	63-135
1,3-dichlorobenzene	64-136	79-120
1,4-dichlorobenzene [2]	69-125	79-116
1,2-dichlorobenzene	71-127	62-143
1,2-dibromo-3-chloropropane	71-128	57-133
1,2,4-trichlorobenzene	52-147	57-142
Xylene (total)	60-140	61-150

Table displays statistical control limits calculated in 2002.

[1] Denotes **Ohio VAP spike compounds. Each recovery must be within established control limits.**

[2] SC DHEC **spike compounds**. Each of these analytes must be recovered within 70-130%.

Attachment 4

COMPUCHEM a division of Liberty Analytical Corp DATE ___/___/___ INITIAL TIME OF TUNE_____ SHIFT/S(A)_____(B)_____(C)____
GC/MS VOLATILE RUN LOG TIME TUNE EXPIRES_____ LINKER /METHOD_____
COMPUCHEM LOGBOOK 11 X 24 (f50052)

PREVENTIVE MAINTENANCE_____

	RETRIEVED	FILE NAME	STORED	pH	Vial	DATE	TIME	Client ID	SDG#	INJ. VOL.*	DF+	CHEMIST	COMMENTS(ETC.)/ DISPOSITION
1						/ /							
2						/ /							
3						/ /							
4						/ /							
5						/ /							
6						/ /							
7						/ /							
8						/ /							
9						/ /							
10						/ /							
11						/ /							
12						/ /							
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16						/ /							
17						/ /							
18						/ /							
19						/ /							
20						/ /							
21						/ /							
22						/ /							
23						/ /							
24						/ /							

*On-column sample aliquot
+Not applicable to soil matrix

SUPERVISOR APPROVAL_____ Date_____

Tune (ID #7008) Lot No. _____

Calibration Group Code / Lot No. _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

10/26/05:jad

Attachment 5

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30431 Lot No.: A027506
Description: 502.2 CAL 2000 Mega-Mix
Expiration Date[1]: August 2005 Storage: Freezer

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1,1-dichloroethane	75-34-3	99%	2,000 µg/ml	+/- .04%
1,1-dichloroethene	75-35-4	99%	2,000 µg/ml	+/- .04%
1,1-dichloropropene	563-58-6	99%	2,000 µg/ml	+/- .04%
1,1,1-trichloroethane	71-55-6	99%	2,000 µg/ml	+/- .04%
1,1,1,2-tetrachloroethane	630-20-6	99%	2,000 µg/ml	+/- .04%
1,1,2-trichloroethane	79-00-5	99%	2,000 µg/ml	+/- .04%
1,1,2,2-tetrachloroethane	79-34-5	99%	2,000 µg/ml	+/- .04%
1,2-dibromo-3-chloropropane	96-12-8	97%	2,000 µg/ml	+/- .04%
1,2-dibromoethane	106-93-4	99%	2,000 µg/ml	+/- .04%
1,2-dichlorobenzene	95-50-1	99%	2,000 µg/ml	+/- .04%
1,2-dichloroethane	107-06-2	99%	2,000 µg/ml	+/- .04%
1,2-dichloropropane	78-87-5	99%	2,000 µg/ml	+/- .04%
1,2,3-trichlorobenzene	87-61-6	99%	2,000 µg/ml	+/- .04%
1,2,3-trichloropropane	96-18-4	99%	2,000 µg/ml	+/- .04%
1,2,4-trichlorobenzene	120-82-1	99%	2,000 µg/ml	+/- .04%
1,2,4-trimethylbenzene	95-63-6	99%	2,000 µg/ml	+/- .04%
1,3-dichlorobenzene	541-73-1	99%	2,000 µg/ml	+/- .04%
1,3-dichloropropane	142-28-9	99%	2,000 µg/ml	+/- .04%
1,3,5-trimethylbenzene	108-67-8	99%	2,000 µg/ml	+/- .04%
1,4-dichlorobenzene	106-46-7	99%	2,000 µg/ml	+/- .04%
2-chlorotoluene	95-49-8	99%	2,000 µg/ml	+/- .04%
2,2-dichloropropane	594-20-7	99%	2,000 µg/ml	+/- .04%
4-chlorotoluene	106-43-4	99%	2,000 µg/ml	+/- .04%
p-isopropyltoluene	99-87-6	99%	2,000 µg/ml	+/- .04%
benzene	71-43-2	99%	2,000 µg/ml	+/- .04%
bromobenzene	108-86-1	99%	2,000 µg/ml	+/- .04%
bromochloromethane	74-97-5	99%	2,000 µg/ml	+/- .04%

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

[1]Expiration date of the unopened ampul stored at recommended temperature.

[2]Listed in alphabetical order.

[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.

[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).

[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

NOTE: Attachment is subject to change without notice.

Attachment 5 (continued)

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS (cont.)

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

PAGE 2

Cat. No.: 30431 Lot No.: A027506

Description: 502.2 CAL 2000 Mega-Mix

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
bromodichloromethane	75-27-4	99%	2,000 µg/ml	+/- .04%
bromoform	75-25-2	99%	2,000 µg/ml	+/- .04%
carbon tetrachloride	56-23-5	99%	2,000 µg/ml	+/- .04%
chlorobenzene	108-90-7	99%	2,000 µg/ml	+/- .04%
chloroform	67-66-3	99%	2,000 µg/ml	+/- .04%
cis-1,2-dichloroethene	156-59-2	99%	2,000 µg/ml	+/- .04%
cis-1,3-dichloropropene	10061-01-5	99%	2,000 µg/ml	+/- .04%
dibromochloromethane	124-48-1	99%	2,000 µg/ml	+/- .04%
dibromomethane	74-95-3	99%	2,000 µg/ml	+/- .04%
ethylbenzene	100-41-4	99%	2,000 µg/ml	+/- .04%
hexachlorobutadiene	87-68-3	99%	2,000 µg/ml	+/- .04%
isopropylbenzene	98-82-8	99%	2,000 µg/ml	+/- .04%
methylene chloride	75-09-2	99%	2,000 µg/ml	+/- .04%
m-xylene	108-38-3	99%	2,000 µg/ml	+/- .04%
naphthalene	91-20-3	99%	2,000 µg/ml	+/- .04%
n-butylbenzene	104-51-8	99%	2,000 µg/ml	+/- .04%
n-propylbenzene	103-65-1	99%	2,000 µg/ml	+/- .04%
o-xylene	95-47-6	99%	2,000 µg/ml	+/- .04%
p-xylene	106-42-3	99%	2,000 µg/ml	+/- .04%
sec-butylbenzene	135-98-8	99%	2,000 µg/ml	+/- .04%
styrene	100-42-5	99%	2,000 µg/ml	+/- .04%
tetrachloroethene	127-18-4	99%	2,000 µg/ml	+/- .04%
tert-butylbenzene	98-06-6	99%	2,000 µg/ml	+/- .04%
toluene	108-88-3	99%	2,000 µg/ml	+/- .04%
trans-1,2-dichloroethene	156-60-5	99%	2,000 µg/ml	+/- .04%
trans-1,3-dichloropropene	10061-02-6	97%	2,000 µg/ml	+/- .04%
trichloroethene	79-01-6	99%	2,000 µg/ml	+/- .04%
Solvent: purge & trap methanol	67-56-1	99%		

[1]Expiration date of the unopened ampul stored at recommended temperature.

[2]Listed in alphabetical order.

[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.

[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).

Attachment 5 (continued)

TCL4 - GASES HIGH

RESTEK

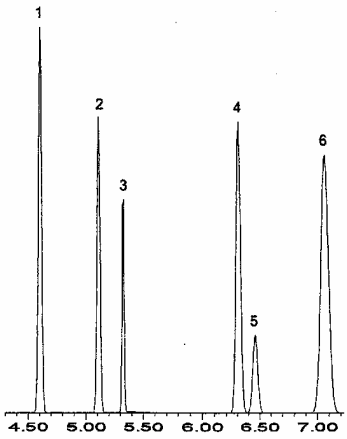
CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30042 Lot No.: A028050
Description: 502.2 Calibration Mix #1
Expiration Date[1]: March 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	Dichlorodifluoromethane	75-71-8	99%	1,997 µg/ml	± 1.9%
2	Chloromethane	74-87-3	99%	2,003 µg/ml	± 1.3%
3	Vinyl chloride	75-01-4	99%	2,003 µg/ml	± 1.4%
4	Bromomethane	74-83-9	99%	1,997 µg/ml	± 1.3%
5	Chloroethane	75-00-3	99%	2,003 µg/ml	± 1.5%
6	Trichlorofluoromethane	75-69-4	99%	2,000 µg/ml	± 0.3%
Solvent:	Purge and trap Methanol	67-56-1	99%		



Column: 105m .32mm 1.8µm
Rtx-502.2 (cat.#10921)
Carrier gas: helium @ 2.2 ml/min.
Temp. program: 50°C (hold 12 min.)
Isothermal
Inj. temp.: 240°C
Det. temp.: 240°C
Detector type: MSD

John Lidgett
John Lidgett—QA Analyst

[1]*Expiration date of the unopened ampul stored at recommended temperature.*
[2]*Listed in alphabetical order.*
[3]*Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.*
[4]*Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*
[5]*Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.*

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

Attachment 5 (continued)

TCL4- KETONES HIGH

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9863-R1
DESCRIPTION: Custom VOA Std #2
LOT #: B3110072
SOLVENT: Methanol

EXPIRATION: Feb 9, 2004

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetone	67-64-1	99.9	1261 ±50.44	1260
Methyl ethyl ketone	78-93-3	100	1261 ±50.44	1261
4-methyl-2-pentanone	108-10-1	99.4	1261 ±50.44	1253
2-hexanone	591-78-6	100	1261 ±50.44	1261
Vinyl acetate	108-05-4	99.9	500.8 ±20.03	500.3
2-Chloroethylvinyl ether	110-75-8	99.7	500.3 ±20.01	498.8
Acrolein	107-02-8	90.0	5575 * ±223.00	5018
Acrylonitrile	107-13-1	99.9	5000 ±200.00	4995

8 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty u_c(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is U_c(y) *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00
Rev. 11/0

Attachment 5 (continued)

TCL4-APPIX H1

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9862-R1
EXPIRATION: Feb 27, 2004
DESCRIPTION: Custom VOA Std #1
LOT #: B3110080 **See reverse for additional certification information.**
SOLVENT: Methanol

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetonitrile	75-05-8	99.9	500.3 ±20.01	499.8
Allyl chloride	107-05-1	99.6	500.0 ±20.00	498.0
1-Chlorohexane	544-10-5	99.0	500.3 ±20.01	495.3
Chloroprene	126-99-8	98	500.0 ±20.00	490.0
trans-1,4-Dichloro-2-butene	110-57-6	97.4	25,670 *±1026.80	25003
p-Dioxane	123-91-1	99.2	25,010 ±1000.40	24810
Ethyl methacrylate	97-63-2	99.0	5010 ±200.40	4960
Isobutyl alcohol	78-83-1	100	25,010 ±1000.40	25010
Methacrylonitrile	126-98-7	99.9	5010 ±200.40	5005
Methyl iodide	74-88-4	100	500.3 ±20.01	500.3
Methyl methacrylate	80-62-6	99.8	5003 ±200.12	4993
Pentachloroethane	76-01-7	97.3	514.0 *±20.56	500.1
Propionitrile	107-12-0	99.9	25,010 ±1000.40	24985

13 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00

Attachment 5 (continued)

RESTEK

CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY—READ MSDS PRIOR TO USE.

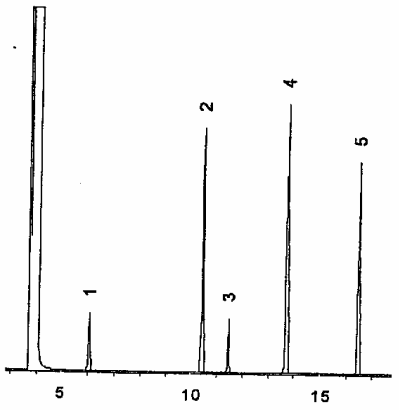
Cat. No.: 30005 Lot No.: A019743

Description: VOA Matrix Spike Mix

Expiration Date[1]: January 2005

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	1,1-dichloroethene	75-35-4	99%	2,500 µg/ml	± 0.2%
2	benzene	71-43-2	99%	2,500 µg/ml	± 0.2%
3	trichloroethene	79-01-6	99%	2,500 µg/ml	± 0.2%
4	toluene	108-88-3	99%	2,500 µg/ml	± 0.2%
5	chlorobenzene	108-90-7	99%	2,500 µg/ml	± 0.2%
Solvent:	purge and trap methanol	67-56-1	99%		

Column: 105m .53mm 3.0µm Rtx®-502.2 (cat.#10910)
Carrier gas: hydrogen @ 40cm/sec
Temp. program: 40°C (hold 2 min.) to 240°C @ 8°C/min. (hold 10 min.)
Inj. temp.: 200°C
Det. temp.: 250°C
Detector type: FID



John Lidgett—QA Analyst

[1]*Expiration date of the unopened ampul stored at recommended temperature.*

[2]*Listed in alphabetical order.*

[3]*As determined by capillary GC/FID unless otherwise noted. Value rounded to the nearest LOWER whole percentage. In addition to GC/FID, chemical identity and purity are confirmed using 2 or more of the following: GC/MS, solid probe MS, GC/ECD, GC/FPD, GC/NPD, GC/TC, HPLC, DSC, FTIR, melting point, refractive index, and Karl Fisher.*

[4]*Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*

[5]*Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.*

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

Attachment 6

Solid and Aqueous LCS Control Limits for 8260B Required by the DoD-QSM

Compound	Aqueous Recovery Limits	Aqueous Marginal Exceedances	Solid Recovery Limits	Solid Marginal Exceedances
Dichlorodifluoromethane	30-155	10-175	70-135	55-145
Chloromethane	40-125	25-140	50-130	40-140
Vinyl chloride	50-145	35-165	11-60	45-140
Bromomethane	30-145	10-165	30-160	10-180
Chloroethane	60-135	50-145	40-155	20-175
Trichlorofluoromethane	60-145	45-160	25-185	10-215
1,1-Dichloroethene	70-130	55-140	65-135	55-150
Carbon disulfide	35-160	15-185	45-160	30-180
Methylene chloride	55-140	40-155	55-140	40-155
trans-1,2-Dichloroethene	60-140	45-150	65-135	55-145
Methyl-tert-butyl ether	65-125	55-135	N/A	N/A
1,1-Dichloroethane	70-135	50-145	75-125	65-135
cis-1,2-Dichloroethene	70-125	60-135	65-125	55-135
2-Butanone	30-150	10-170	30-160	10-180
Chloroform	65-135	50-150	70-125	65-135
1,1,1-Trichloroethane	65-130	55-145	70-135	55-145
Carbon tetrachloride	65-145	55-150	65-135	55-145
Benzene	80-120	75-130	75-125	65-135
1,2-Dichloroethane	70-130	60-140	70-135	60-145
Trichloroethene	70-125	60-135	75-152	70-130
1,2-Dichloropropane	75-125	65-135	70-120	65-125
Bromodichloromethane	75-120	70-130	70-130	60-135
cis-1,3-dichloropropene	70-130	60-140	70-125	65-135
4-Methyl-2-pentanone	60-135	45-145	45-145	30-165
Toluene	75-120	70-130	70-125	60-135
trans-1,3-Dichloropropene	55-140	40-155	65-125	55-140
1,1,2-trichloroethane	75-152	65-135	60-125	50-140
Tetrachloroethene	45-150	25-165	65-140	55-150
2-Hexanone	55-130	45-140	45-145	30-160
Dibromochloromethane	60-135	45-145	65-130	55-140
1,2-Dibromoethane	80-120	75-125	70-125	60-135
Chlorobenzene	80-120	75-130	75-125	65-130
Ethylbenzene	75-125	65-135	75-125	65-135
Styrene	65-135	55-145	75-125	65-135
Bromoform	70-130	60-140	55-135	45-150
Isopropyl benzene	75-125	65-135	75-130	70-140
1,1,2,2-Tetrachloroethane	80-130	75-135	55-130	40-145
1,3-Dichlorobenzene	75-125	65-130	70-125	65-135
1,4-Dichlorobenzene	75-125	65-130	70-125	65-135
1,2-Dichlorobenzene	70-120	60-130	75-120	65-125
1,2-Dibromo-3-Chloropropane	50-130	35-145	40-135	25-150
1,2,4-Trichlorobenzene	65-135	55-145	65-130	55-140

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-238 SOP Section #: 1.1.4.1 Revision #: 4

SOP Title: Effective date: (QA fills in)

Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035 — 1/14/04

- Procedure prepared by: Edward E. Meiers Date: 1/14/04
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Diane C. Ellmore Date: 1/14/04
- Reason for change: Freezing Preservation added
- This procedure meets the requirements of the following approved method references:

US EPA CLP SOW OLM04.3, plus revisions; NELAC Standards, approved May 2001, plus revisions; SW846, Update 3, 3rd Edition, 12/96, Method 5035

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

ORIGINAL

Annual Review—Signature: _____ Date: 8.30.05

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -238: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035

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1.0 Scope and Application

This procedure is used to prepare soil, sediment, and sludge samples for the analysis of volatile organic compounds by the closed-system purge and trap process using GC/MS Method 8260B or CLP. The procedure is based on Method 5035. The prepared sample may also be analyzed for GRO by Method 8015B. Provisions are also included to prepare (and preserve) samples with methanol when higher concentrations of volatiles are present in the sample.

Method detection limits and reporting limits are found in the respective analytical SOPs for the methods described in the above paragraph.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Several options are available when EnCore sampling devices are utilized. These incorporate the use of a sodium bisulfate preservative, freezing at a temperature colder than –7 °C, or, for samples containing high levels of target anlaytes, preserving in methanol.

Sodium Bisulfate Preservation

A five (5) gram sample is taken in the field using a disposable EnCore sampling device. At the laboratory and within forty-eight (48) hours of sampling, the sample is weighed into a standard forty (40) milliliter volatile vial containing a magnetic stirring bar and five (5) mL of a sodium bisulfate solution (0.1 g of sodium bisulfate per gram of sample weighed.) The vial is sealed with a screw-top, PTFE-lined, septum-sealed cap. The sample container is stored at 4°C ± 2°C until analysis, which must be completed within 14 days of collection.

Freezing Preservation

A five (5) gram sample is taken in the field using a disposable EnCore sampling device. At the laboratory and within forty-eight (48) hours of sampling, the contents of the EnCore sampling device are placed into a tared, dry, closed-system purge-and-trap vial, re-weighed to obtain the final sample weight, and the vial placed into a freezer maintained at a temperature colder than –7 °C. This option can be performed with or without the addition of 5 mL of reagent water.

For the State of Florida DEP samples may be frozen to –10 °C but only after the entire contents of the sampling device are extruded into the sample analysis vial containing reagent water.

Methanol Preservation

For screening purposes, and also to provide a mechanism to determine the concentration of target analytes which may be present at high levels, the contents of another 5 gram EnCore sampler are also weighed and placed into a vial containing methanol.

For some projects, a 25 gram EnCore sampler may be used and its contents are placed in a 2 oz. jar and preserved with methanol.

Depending on specific project requirements, samples may be received by the laboratory already in sealed 40 mL vials, containing a stirring bar and sodium bisulfate solution, or preserved with methanol.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

For CLP the reporting limit is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/Kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.5 GRO – Gasoline Range Organics

4.0 Interferences

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks.

4.2 The use of non-PTFE plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging devise must be avoided.

4.3 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank carried through sampling and storage serves as a check of contamination.

4.4 The laboratory where volatiles are prepared and analyzed should be completely free of solvents. Persons leaving the organic sample preparation laboratory must not enter the volatile laboratory until sufficient time has passed to avoid the introduction of solvent vapors.

5.0 Safety

5.1 When following this procedure, be sure to wear protective gloves, lab coats, safety glasses and work under a hood.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Sample containers - standard forty (40) milliliter volatile vial with screw-top, PTFE-lined, septum-sealed cap. Vials are purchased pre-cleaned from certifying vendors. **Additionally, these closed-system purge-and-trap vials may be purchased, each containing 0.2 g of sodium bisulfate and 5 mL of organic-free water.**

6.2 Magnetic stirring bar - PTFE or glass coated.

6.3 Stainless steel spatulas.

6.4 Top-loading balance, capable of weighing accurately to 0.01 g

6.5 Five (5) or twenty-five (25) gram disposable EnCore sampling device.

6.6 Graduated pipettes capable of delivering 1-10 mL.

7.0 Reagents & Standards

Standard preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.)

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remained of this SOP as DI water.

7.2 Methanol – purge and trap grade or equivalent.

7.3 Sodium bisulfate (NaHSO4) ACS reagent grade.

7.3.1 20% sodium bisulfate solution – 200 g $NaHSO_4$ in 1000 ml DI water.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 A method blank is prepared for every preparation batch following steps 11.3 - 11.5, and consists of a purified solid matrix, i.e. Ottawa sand.

9.2 Steps 11.3 - 11.5 **(or steps 11.10 or 11.11, depending on the option exercised)** are repeated twice more from a designated sample and used for the matrix spike (MS) and matrix spike duplicate (MSD). An additional 5-g portion of a purified solid matrix (Ottawa sand) is weighed into a separate sample container for the blank spike (BS). This is called a matrix spike blank for the NYSASP. A BS is prepared with each preparation batch.

9.3 Duplicates, at a frequency of 10%, are required when processing samples submitted to meet the regulatory requirements of North Carolina.

10.0 Calibration & Standardization

10.1 Balance Calibration

10.1.1 Ensure the balance is calibrated for the day prior to weighing samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Glassware must be scrupulously clean. The glassware and magnetic stir bars are washed with hot soapy water, rinsed with hot water and finally rinsed with laboratory pure water. The glassware is dried at 105 ± 5°C for one (1) hour or overnight in an oven. If the glassware has been stored in the laboratory environment, place it in oven for one (1) hour (105 ± 5°C) and allow it to cool in a contaminant free environment before using. Alternatively, rinse thoroughly with DI water before use.

The 40 mL VOA vials are purchased pre-cleaned by certifying vendors. **These vials can also be purchased already containing the sodium bisulfate solution.**

11.2 When the samples have come to room temperature and are ready to be prepared, assemble the designated materials in the hood.

11.3 For each sample, label (with permanent ink typically on white tape) three (3) standard volatile vial sample containers with the laboratory ID number of the sample.

11.4 Open the five (5) gram disposable EnCore sampling device. Do not discard any supernatant liquid.

11.5 Place a vial (with cap), containing a magnetic stirring bar, on the top loading balance, and add a 5 ml aliquot of $NaHSO_4$ solution (7.3.1). **Alternatively, closed-system purge-and-trap vials already containing the sodium bisulfate solution may be used.** The weight is recorded on the EnCore Preparation Worksheet (attachment 1). Add the contents of the EnCore sampler to the vial. Read and record weight. Seal the volatile vial with the PTFE-lined screw cap.

11.6 Prepare a back-up aliquot by repeating step 11.4 and 11.5.

11.7 Place another 40 ml vial and cap on the top-loading balance and transfer 5.0 ml ± 0.1 ml of methanol. Record the weight. Transfer the contents of a 5 gram EnCore sampler to the vial. Seal the vial with the PTFE-lined screw cap and record the weight. Use this sample for screening purposes and medium/high level analysis, if necessary.

11.8 Repeat steps 11.4-11.8 until the required number of samples are prepared, completing the worksheet as the preparation is accomplished.

11.9 If, during the addition of a sample to a vial containing the sodium bisulfate solution, effervescing occurs, that sample should be properly discarded. Another vial should be obtained, weighed, the contents of a 5 gram EnCore sample added and the vial should be sealed with a PTFE-lined screw cap and the weight recorded. This sample should be placed in a freezer at **a tempeature colder than –7 °C.** Pre-testing for effervescence can also be performed using the bottle supplied for percent moisture.

11.10 Sample preservation by freezing, as described in step 11.9, is also a viable option for all samples received in EnCore sampling devices. The contents of the EnCore sampling devices are placed in a tared, dry, closed-system purge-

and-trap device, re-weighed to obtain the final sample weight, and the vial placed into a freezer maintained at a temperature colder than –7 °C.

11.11 Another option permits the contents of the EnCore sampling device to be extruded into a tared close-system purge-and-trap device containing 5 mL of reagent water. When this option is exercised, the sealed vial containing the sample and water should be placed in the freezer (colder than –7 °C) in a horizontal manner so the vial will not burst upon freezing.

11.12 Samples prepared by this method are now ready for analysis by the appropriate analytical method. When the set is complete, store the samples in the appropriate volatile GC or GC/MS laboratory refrigerator until analysis. The refrigerator temperature must be at 2°-4.4°C in order to meet NCDENR storage requirements.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst (Attachment 2). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP Statement of Work for Organics, Multi-Media, Multi-Concentration, OLM04.3, plus revisions

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Method 5035, **Draft Method 5035A, and** Method 8260B

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 New York State Analytical Services Protocol (NYSASP), June, 2000, plus revisions

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **approved May 2001**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision **4**, 12/**10**/**03**, plus revisions

16.13 Sample Preparation Procedure –143, "% Moisture Determination (Undecanted) (EPA CLP SOW, SW8746, and NYSASP", SOP Section 2.8.1.

16.14 Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples."

16.15 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance"

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – EnCore Preparation Worksheet

17.2 Attachment 2 – Single Analyst Capability Study

Attachment 1

COMPUCHEM
a Division of Liberty Analytical
EnCore™ Preparation Worksheet
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SDG:_____ Prepared By:_____ Date:_____

	LoginNum	Date	Time	Weight of Jar (g) *	Weight of Jar + Sample (g)	Weight of Sample (g)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						

Volume of Methanol Added:_____

Lot Numbers of Preservatives: Sodium Bisulfate:_____ Methanol:_____

Reviewed By:_____

*Weight of Jar for Sodium Bisulfate preserved Encores is the sum of the weights of the vial, preservative and stir bar
*Weight of Jar for Methanol preserved Encores is the sum of the weights of the vial and preservative

Note: The insertion of "N/A" for "Volume of Methanol Added" and "Lot Numbers of Preservatives: Sodium Bisulfate:____ Methanol:_____" indicates a freezing option was employed.

dce:1/14/2004

Attachment 2

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: Jeremy Smith
Study Date: April 18, 2000
Method: 5035/8260B, 5gm Soil
Instrument/Column/Detector:
f50052

Compounds	TrueVal ug/kg	Rep #1 ug/kg	Rep #2 ug/kg	Rep #3 ug/kg	Rep #4 ug/kg	Mean ug/kg	Mean % R	EPA %R	SD(n-1) ug/kg	EPA %RSD	-3SD of (x)	+3SD of (x)	-3SD % R	+3SD %R	RSD %
Dibromofluoromethane	50	55.16	54.72	50.24	54.07	53.55	107	NA	2.25	NA	46.8	60.3	87	113	4.2
1,2-Dichloroethane-d4	50	55.39	56.72	52.84	54.28	54.81	110	NA	1.65	NA	49.9	59.8	91	109	3.0
Toluene-d8	50	48.85	49.86	45.84	48.42	48.24	96	NA	1.71	NA	43.1	53.4	89	111	3.5
Bromofluorobenzene	50	48.68	48.32	42.59	45.19	46.20	92	NA	2.87	NA	37.6	54.8	81	119	6.2
Chloromethane	50	48.03	46.52	41.81	45.74	45.53	91	NA	2.65	NA	37.6	53.5	83	117	5.8
Vinyl Chloride	50	46.69	44.65	38.97	44.32	43.66	87	34.2	3.30	13.0	33.8	53.5	77	123	7.5
Bromomethane	50	53.44	53.89	47.79	51.74	51.72	103	NA	2.78	NA	43.4	60.0	84	116	5.4
Chloroethane	50	60.40	59.08	53.29	58.47	57.81	116	NA	3.12	NA	48.5	67.2	84	116	5.4
1,1-Dichloroethene	50	53.53	55.28	48.78	52.65	52.56	105	79.2	2.75	5.7	44.3	60.8	84	116	5.2
Acetone	130	145.6	145.1	149.7	147.5	147.0	113	NA	2.08	NA	140.7	153.2	96	104	1.4
Carbon disulfide	50	53.52	55.46	49.62	53.18	52.95	106	NA	2.43	NA	45.6	60.2	86	114	4.6
Methylene Chloride	50	59.07	57.92	51.39	56.62	56.25	113	107	3.39	9.1	46.1	66.4	82	118	6.0
trans-1,2-Dichloroethene	50	54.97	55.73	49.14	53.05	53.22	106	104	2.95	0.7	44.4	62.1	83	117	5.5
Methyl tert butyl ether	50	58.46	59.68	54.58	56.71	57.36	115	NA	2.22	NA	50.7	64.0	88	112	3.9
1,1-Dichloroethane	50	51.63	54.30	47.29	50.78	51.00	102	84.4	2.89	6.4	42.3	59.7	83	117	5.7
cis-1,2-Dichloroethene	50	54.79	57.23	49.56	52.02	53.40	107	113	3.33	9	43.4	63.4	81	119	6.2
2-Butanone	130	128.5	124.5	126.4	132.0	127.9	98	NA	3.21	NA	118.3	137.5	92	108	2.5
Chloroform	50	55.08	58.27	51.15	54.26	54.69	109	116	2.93	12.2	45.9	63.5	84	116	5.4
1,1,1-Trichloroethane	50	51.59	53.51	47.34	51.33	50.94	102	117	2.59	21.2	43.2	58.7	85	115	5.1
Carbon tetrachloride	50	56.22	59.49	53.77	58.58	57.02	114	112	2.56	9.4	49.3	64.7	87	113	4.5
1,2-Dichloroethane	50	56.49	57.33	52.43	56.12	55.59	111	NA	2.17	NA	49.1	62.1	88	112	3.9
Benzene	50	49.77	52.32	44.86	48.70	48.91	98	103	3.10	11.2	39.6	58.2	81	119	6.3
Trichloroethene	50	53.47	55.09	48.43	50.17	51.79	104	94.6	3.03	12.7	42.7	60.9	82	118	5.9
1,2-Dichloropropane	50	51.36	52.93	46.55	48.84	49.92	100	117	2.81	10.5	41.5	58.3	83	117	5.6
Bromodichloromethane	50	56.84	59.52	52.46	55.27	56.02	112	117	2.95	13.1	47.2	64.9	84	116	5.3
cis-1,3-Dichloropropene	50	54.47	57.15	48.88	50.81	52.83	106	NA	3.70	NA	41.7	63.9	79	121	7.0
4-Methyl-2-pentanone	130	122.1	116.4	120.0	127.6	121.5	93	NA	4.68	NA	107.5	135.6	88	112	3.9
Toluene	50	48.19	49.79	45.38	48.02	47.85	96	118	1.83	16.9	42.4	53.3	89	111	3.8
trans-1,3-Dichloropropene	50	53.69	54.96	51.11	52.83	53.15	106	NA	1.62	NA	48.3	58.0	91	109	3.0
1,1,2-Trichloroethane	50	50.80	51.03	48.82	54.06	51.18	102	111	2.16	12.1	44.7	57.7	87	113	4.2
Tetrachloroethene	50	51.16	52.03	48.45	51.95	50.90	102	NA	1.68	NA	45.9	55.9	90	110	3.3
2-Hexanone	130	120.6	119.6	119.7	122.5	120.6	93	NA	1.36	NA	116.5	124.7	97	103	1.1
Dibromochloromethane	50	56.73	57.40	52.38	56.46	55.74	111	118	2.28	12.5	48.9	62.6	88	112	4.1
Chlorobenzene	50	52.07	52.48	47.87	49.63	50.51	101	99.3	2.16	15	44.0	57.0	87	113	4.3
Ethylbenzene	50	50.75	52.58	46.32	47.34	49.25	98	112	2.92	17.5	40.5	58.0	82	118	5.9
m,p-Xylene	100	97.59	104.76	90.44	94.19	96.75	97	98.5	6.09	15.7	78.5	115.0	81	119	6.3
o-Xylene	50	50.15	52.09	45.95	46.33	48.63	97	103	2.99	17.3	39.7	57.6	82	118	6.1
Styrene	50	48.39	52.51	44.56	45.84	47.83	96	101	3.51	15.7	37.3	58.3	78	122	7.3
Bromoform	50	57.64	60.05	56.53	61.67	58.97	118	122	2.32	9.9	52.0	65.9	88	112	3.9
1,1,1,2-Tetrachloroethane	50	55.25	53.05	49.84	57.87	54.00	108	NA	3.40	NA	43.8	64.2	81	119	6.3
1,2-Dichloroethene (total)	100	109.8	112.6	98.6	105.3	106.6	107	NA	6.11	NA	88.2	124.9	83	117	5.7
Xylene (total)	150	161.7	171.8	149.3	154.0	159.2	106	NA	9.84	NA	129.7	188.7	81	119	6.2

APPENDIX E-2

Standard Operating Procedures
For Laboratory Analyses – Soil Vapor

TO-13A

7.0 TO-13A AND 8270C – SEMIVOLATILE COMPOUNDS

This method involves GC/MS full scan or SIM mode analysis of semi-volatile organic compounds in ambient air samples collected on PUF/XAD2 cartridges. In relation to the prescribed media, sampling and collection efficiency for compounds not listed in TO-13A has not been evaluated. Samples are prepared by either soxhlet or Pressurized Fluid Extraction (PFE) by EPA Method 3545A and analyzed for Polynuclear Aromatic Hydrocarbons (PAHs) using a quadrupole GC/MS in full scan or SIM mode by TO-13A protocol. In addition, the target compound list is often extended to include analysis of Method 8270 semi-volatile compounds. Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Air Toxics Ltd. also performs semi-volatile analysis by SW-846 Method 8270C. The extraction process of MM5 trains follows SW846 Method 3542, and the QC criteria differ from Method TO-13A analysis. The QC criteria and QC summary tables for Method 8270C analysis are in the section following the TO-13A tables.

Table 7-1 Summary of Method Modifications for TO-13A

Requirements	EPA Method TO-13A	Air Toxics Ltd. Modifications
Extraction Solvent	10% ether in hexane for PUF; DCM for XAD sorbent. Final extract in hexane.	DCM for PUF/XAD cartridge and XAD sorbent. Final extract in DCM.
Glassware Cleaning	Muffle furnace is utilized.	Solvent cleaning procedure is used.
Extraction Technique	Soxhlet extraction.	Soxhlet extraction or pressurized fluid extraction (PFE).
Reporting List	19 PAHs.	See Tables 7-2 & 7-3.
Calibration range:	0.1-2.5 µg/mL in Hexane	1.0-160 µg/mL in Methylene chloride for quad or 0.1-40 µg/mL for SIM.
Surrogate	Field surrogates: Fluoranthene-d10 and Benzo(a)pyrene-d12.	Field surrogates: provided upon request.
Solvent Process Blank	One each analytical batch.	Not performed: each solvent lot is certified.
Method Blank	< MDL.	<Reporting Limit.

Table 7-2 Modified Method TO-13A

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
2-Chloronaphthalene*	0.1	1.0	NA	≤ 30	± 50	± 30	≤ 25%
2-Methylnaphthalene*	0.1	1.0	NA	≤ 30	± 50	± 30	≤ 25%
Acenaphthylene	0.1	1.0	1.3	≤ 30	± 50	± 30	≤ 25%
Acenaphthene	0.1	1.0	0.8	≤ 30	± 50	± 30	≤ 25%
Anthracene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Benzo(a)anthracene	0.1	1.0	0.8	≤ 30	± 50	± 30	≤ 25%
Benzo(e)pyrene*	0.1	1.0	NA	≤ 30	± 50	± 30	≤ 25%
Benzo(a)pyrene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Benzo(b)fluoranthene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Benzo(g,h,i)perylene	0.1	1.0	0.5	≤ 30	± 50	± 30	≤ 25%
Benzo(k)fluoranthene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Chrysene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Dibenz(a,h)anthracene	0.1	1.0	0.4	≤ 30	± 50	± 30	≤ 25%
Fluoranthene	0.1	1.0	0.6	≤ 30	± 50	± 30	≤ 25%
Fluorene	0.1	1.0	0.9	≤ 30	± 50	± 30	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.1	1.0	0.5	≤ 30	± 50	± 30	≤ 25%
Naphthalene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Phenanthrene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Pyrene	0.1	1.0	0.6	≤ 30	± 50	± 30	≤ 25%

*** Not included in the TO-13A method.**

The following two compounds can be analyzed upon client's request.

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
Perylene	NA	1.0	0.5	≤ 30	± 50	± 30	≤ 25%
Coronene	NA	1.0	0.7	≤ 30	± 50	± 30	≤ 25%

Table 7-3 Modified Method TO-13A-Extended

Analyte	Minimum ICAL RRF	RL (µg)	ICAL [1] (%RSD)	ISCV (%R)	Precision %RPD
1,2,4-Trichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,2-Dichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,3-Dichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,4-Dichlorobenzene - CCC	NA	1.0	≤ 30	± 50	≤ 25%
2,4,5-Trichlorophenol	NA	5.0	≤ 30	± 50	≤ 25%
2,4,6-Trichlorophenol - CCC	NA	5.0	≤ 30	± 50	≤ 25%
2,4-Dichlorophenol - CCC	NA	5.0	≤ 30	± 50	≤ 25%

Analyte	Minimum ICAL RRF	RL (µg)	ICAL [1] (%RSD)	ISCV (%R)	Precision %RPD
2,4-Dimethylphenol	NA	5.0	≤ 30	± 50	≤ 25%
2,4-Dinitrophenol - SPCC	0.05	20	≤ 30	± 50	≤ 25%
2,4-Dinitrotoluene	NA	5.0	≤ 30	± 50	≤ 25%
2,6-Dinitrotoluene	NA	5.0	≤ 30	± 50	≤ 25%
2-Chloronapthalene	NA	1.0	≤ 30	± 50	≤ 25%
2-Chlorophenol	NA	5.0	≤ 30	± 50	≤ 25%
2-Methylnapthalene	NA	1.0	≤ 30	± 50	≤ 25%
2-Methylphenol	NA	5.0	≤ 30	± 50	≤ 25%
2-Nitroaniline	NA	10	≤ 30	± 50	≤ 25%
2-Nitrophenol – CCC	NA	5.0	≤ 30	± 50	≤ 25%
3,3-Dichlorobenzidine	NA	20	≤ 30	± 50	≤ 25%
3-Nitroaniline	NA	10	≤ 30	± 50	≤ 25%
4,6-Dinitro-2-methylphenol	NA	10	≤ 30	± 50	≤ 25%
4-Bromophenyl-phenyl ether	NA	1.0	≤ 30	± 50	≤ 25%
4-Chloro-3-methylphenol - CCC	NA	5.0	≤ 30	± 50	≤ 25%
4-Chloroaniline	NA	10	≤ 30	± 50	≤ 25%
4-Chlorophenyl-phenyl ether	NA	1.0	≤ 30	± 50	≤ 25%
4-Methylphenol	NA	5.0	≤ 30	± 50	≤ 25%
4-Nitroaniline	NA	10	≤ 30	± 50	≤ 25%
4-Nitrophenol – SPCC	0.05	20	≤ 30	± 50	≤ 25%
Acenaphthylene	1.3	1.0	≤ 30	± 50	≤ 25%
Acenaphthene – CCC	0.8	1.0	≤ 30	± 50	≤ 25%
Anthracene	0.7	1.0	≤ 30	± 50	≤ 25%
Benzo(a)anthracene	NA	1.0	≤ 30	± 50	≤ 25%
Benzo(a)pyrene - CCC	0.7	1.0	≤ 30	± 50	≤ 25%
Benzo(e)pyrene	0.5	1.0	≤ 30	± 50	≤ 25%
Benzo(b)fluoranthene	0.7	1.0	≤ 30	± 50	≤ 25%
Benzo(g,h,i)perylene	NA	1.0	≤ 30	± 50	≤ 25%
Benzo(k)fluoranthene	NA	1.0	≤ 30	± 50	≤ 25%
Benzoic Acid	NA	30	≤ 30	± 50	≤ 25%
Bis(2-Chloroethoxy) Methane	NA	1.0	≤ 30	± 50	≤ 25%
Bis(2-Chloroispropyl) Ether	NA	1.0	≤ 30	± 50	≤ 25%
Bis(2-Chlroethyl) Ether	NA	1.0	≤ 30	± 50	≤ 25%
Bis(2-Ethylhexyl)phthalate	NA	5.0	≤ 30	± 50	≤ 25%
Butylbenzylphthalate	NA	5.0	≤ 30	± 50	≤ 25%
Chrysene	0.7	1.0	≤ 30	± 50	≤ 25%
di-n-Butylphthalate	NA	5.0	≤ 30	± 50	≤ 25%
di-n-Octylphthalate - CCC	NA	5.0	≤ 30	± 50	≤ 25%
Dibenz(a,h)anthracene	0.4	1.0	≤ 30	± 50	≤ 25%
Dibenzofuran	NA	1.0	≤ 30	± 50	≤ 25%
Diethylphthalate	NA	5.0	≤ 30	± 50	≤ 25%
Dimethylphthalate	NA	5.0	≤ 30	± 50	≤ 25%
Fluoranthene – CCC	0.6	1.0	≤ 30	± 50	≤ 25%
Fluorene	0.9	1.0	≤ 30	± 50	≤ 25%

Analyte	Minimum ICAL RRF	RL (µg)	ICAL [1] (%RSD)	ISCV (%R)	Precision %RPD
Hexachlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
Hexachlorobutadiene - CCC	NA	1.0	≤ 30	± 50	≤ 25%
Hexachlorocylcopentadiene-SPCC	0.05	20	≤ 30	± 50	≤ 25%
Hexachloroethane	NA	1.0	≤ 30	± 50	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.5	1.0	≤ 30	± 50	≤ 25%
Isophorone	NA	1.0	≤ 30	± 50	≤ 25%
n-Nitroso-di-n-propylamine–SPCC	0.05	1.0	≤ 30	± 50	≤ 25%
n-Nitrosodiphenylamine – CCC	NA	10	≤ 30	± 50	≤ 25%
Naphthalene	0.7	1.0	≤ 30	± 50	≤ 25%
Nitrobenzene	NA	1.0	≤ 30	± 50	≤ 25%
Pentachlorophenol – CCC	NA	20	≤ 30	± 50	≤ 25%
Phenanthrene	0.7	1.0	≤ 30	± 50	≤ 25%
Phenol – CCC	NA	5.0	≤ 30	± 50	≤ 25%
Pyrene	0.6	1.0	≤ 30	± 50	≤ 25%

[1] With 10% exception not to exceed 40%

Table 7-4 Surrogates (Full Scan)

Analyte	(%R)
2,4,6-Tribromophenol	50 – 150
2-Fluorophenol	50 – 150
Nitrobenzene-d_5	50 – 150
Phenol-d_5	50 – 150
Fluorene-d10	60 – 120
Pyrene-d10	60 – 120

Table 7-5 Internal Standards

Analyte	(%)
Acenaphthene-d_{10}	50 – 200
Chrysene-d_{12}	50 – 200
1,4-Dichlorobenzene-d_4	50 – 200
Naphthalene-d_8	50 – 200
Perylene-d_{12}	50 – 200
Phenanthrene-d_{10}	50 – 200

Table 7-6 TO-13A-Surrogates (Standard and SIM)

Analyte	Accuracy (% R)*
Fluorene-d_{10}	60 - 120
Pyrene-d_{10}	60 - 120

Table 7-7 Extracted Laboratory Control Spikes for Modified TO-13A-Extended

Analyte	(%R)
1,2,4-Trichlorobenzene***	50 – 150
1,4-Dichlorobenzene***	50 – 150
2,4-Dinitrotoluene***	50 – 150
2-Chlorophenol***	50 – 150
4-Chloro-3-methylphenol***	50 – 150
4-Nitrophenol***	50 – 150
Acenaphthene*	60 – 120
N-Nitroso-di-n-propylamine***	50 – 150

Pentachlorophenol**	22 – 109
Phenol***	50 – 150
Pyrene*	60 – 120

* *The LCS and Surrogate limits are derived from Compendium Method TO-13A Sections 13.3.7.4 and 13.4.6.3 January, 1999. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 – 150 % are applied.*

** *Pentachlorophenol is not included in Compendium Method TO-13A and has been shown to be erratically recovered from XAD media therefore historical Control Limits are used. Limits are updated periodically as needed.*

*** *Compounds outside of the TO-13A method*

Table 7-8 Extracted Laboratory Control Samples for TO-13A (PAHs) in Full Scan and SIM

Analyte	(%R)
Napthalene	60 – 120
Acenapthylene	60 – 120
Acenaphtene	60 – 120
Flourene	60 – 120
Phenanthrene	60 – 120
Anthracene	60 – 120
Fluoranthene	60 – 120
Pyrene	60 – 120
Benzo (a) anthracene	60 – 120
Chrysene	60 – 120
Benzo (b) flouranthene	60 – 120
Benzo (k) flouranthene	60 – 120
Benzo (a) pyrene	60 – 120
Indeno (1,2,3-cd) pyrene	60 – 120
Dibenzo (a,h) anthracene	60 – 120
Benoz (g,,h,i) perylene	60 – 120

Table 7-9 Summary of Calibration and QC Procedures for EPA Method TO-13A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hrs.	SW-846 tuning criteria for semivolatiles analysis. DDT% Breakdown< 20%	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	ICAL criteria in tables 7-2 and 7-3.	Correct problem then repeat initial calibration.
ICAL LCS	All analytes – Once per initial calibration.	All target compound recoveries must be between 50 – 150%.	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock immediately after the DFTPP tune check.	PAHs list: meet min. RRF requirement PAHs list/ short list %D ≤ 30% Semivol full list: SPCCs: RF ≥ 0.050 %D ≤ 30% with 10% exception not to exceed 40%. Flag all results outside of compliance with the exception of high bias associated with non-detects.	Investigate and correct the problem, up to and including re-calibration if necessary. High bias associated with non-detects in samples will not result in re-analysis.
Internal Standards (IS)	As each standard, blank, and sample is being aliquoted.	For CCV: Area count within 50 to 200% of the mid point of ICAL. For blanks, samples and non-CCV QC Checks: retention times within ± 0.33 minutes (20 seconds) and area counts within 50 to 200% of the CCV.	For CCVs: Investigate and correct the problem before proceeding with sample analysis. If interferences are present, a secondary ion may be selected. For blanks: inspect the system and re-analyze the blank. For samples and non-CCV QC: unless there is obvious matrix effect, re-analyze the samples and dilute the sample until the IS meet the criteria, narrate the data to indicate interference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogates	With all samples and blanks prior to extraction.	See Table 7-4.	A new aliquot of the extract is analyzed. If Surrogate recoveries are out-of-control a second time, data is flagged and narrated. Re-analysis is not necessary for obvious matrix effects (data is flagged for out-of-control surrogate recoveries). Air samples cannot be re-extracted.
Extracted LCS	With each set of up to 20 extracted samples.	See LCS Criteria in tables 7-7 and 7-8.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.
Laboratory Blank	With each set of up to 20 extracted samples.	Results less than laboratory reporting limit.	Flag the data.
Solvent Blanks	When samples that are extracted together are analyzed on different analytical shifts.	All target compounds below the reporting limit.	Flag the data.
Laboratory Duplicates	10% of the samples.	RPD ≤ 25% for all hits > 5X RLs.	Narrate the data.

Table 7-10 Summary of Method Modifications for EPA Methods 3510/3542 and 8270C

Requirements	EPA Method 8270C	Air Toxics Ltd. Modifications
Linearity of ICAL	Use mean RF for non-CCC compounds if %RSD≤ 15%. If %RSD>15%, use a) linear regression equation that does not pass through the origin. R >/= 0.99, or b) non-linear (i.e., 6 points for a quadratic model).	Use mean RF for non-CCC compounds when %RSD≤ 30%.
RT for CCV	Within +/- 30 seconds of the mid-point standard from the initial curve.	Frequent column maintenance results in RT shift; therefore this requirement is not practical.

Table 7-11 SW-846 Modified Method 8270C Standard Analyte List

Analyte	RL (µg)	Acceptance Criteria			
		ICAL (%RSD)[1]	ISCV (%R)[2]	CCV[3]	Precision\% RPF
1,2,4-Trichlorobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
1,2-Dichlorobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
1,3-Dichlorobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
1,4-Dichlorobenzene - CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
2,4,5-Trichlorophenol	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2,4,6-Trichlorophenol - CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
2,4-Dichlorophenol - CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
2,4-Dimethylphenol	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2,4-Dinitrophenol - SPCC	20	≤ 15	± 50	**RF > 0.050**	≤ 25%
2,4-Dinitrotoluene	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2,6-Dinitrotoluene	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2-Chloronaphthalene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2-Chlorophenol	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2-Methylnaphthalene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2-Methylphenol	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
2-Nitroaniline	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
2-Nitrophenol – CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
3,3-Dichlorobenzidine	20	≤ 15	± 50	%D ≤ 20%	≤ 25%
3-Nitroaniline	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
4,6-Dinitro-2-methylphenol	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
4-Bromophenyl-phenyl ether	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
4-Chloro-3-methylphenol - CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
4-Chloroaniline	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
4-Chlorophenyl-phenyl ether	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
4-Methylphenol	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
4-Nitroaniline	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
4-Nitrophenol – SPCC	20	≤ 15	± 50	**RF > 0.050**	≤ 25%
Acenaphthylene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Acenaphthene – CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Anthracene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Benzo(a)anthracene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Benzo(a)pyrene - CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Benzo(b)fluoranthene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Benzo(g,h,i)perylene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Benzo(k)fluoranthene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Benzoic Acid	30	≤ 30	± 50	%D ≤ 20%	≤ 25%
Bis(2-Chloroethoxy) Methane	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Bis(2-Chloroispropyl) Ether	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Bis(2-Chlroethyl) Ether	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Bis(2-Ethylhexyl)phthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Butylbenzylphthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%

Analyte	RL (µg)	Acceptance Criteria			
		ICAL (%RSD)①	ISCV (%R)②	CCV③	Precision\% RPF
Chrysene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
di-n-Butylphthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
di-n-Octylphthalate - CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Dibenz(a,h)anthracene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Dibenzofuran	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Diethylphthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Dimethylphthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Fluoranthene – CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Fluorene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Hexachlorobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Hexachlorobutadiene – CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Hexachlorocylcopentadiene – SPCC	20	≤ 15	± 50	**RF > 0.050**	≤ 25%
Hexachloroethane	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Indeno(1,2,3-c,d)pyrene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Isophorone	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
n-Nitroso-di-n-propylamine – SPCC	1.0	≤ 15	± 50	**RF > 0.050**	≤ 25%
n-Nitrosodiphenylamine – CCC	10	≤ 30	± 50	%D ≤ 20%	≤ 25%
Naphthalene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Nitrobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Pentachlorophenol – CCC	20	≤ 30	± 50	%D ≤ 20%	≤ 25%
Phenanthrene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Phenol – CCC	5.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Pyrene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%

① *Can use the mean RSD criterion of ≤ 15% as noted in par. 7.5.1.2.1 of SW-846, 8000B.*

② *No more than 10% of the target compounds are allowed to exceed the limit.*

③ *If %D for all CCC is less than or equal to 20%, then the CCV is assumed to be valid. If the CCCs are not included in the list of analytes for a project, then all analytes must meet the 20% D.*

Table 7-12 Surrogates

Analyte	Accuracy④ (% R)
2,4,6-Tribromophenol	10 – 123
2-Fluorobiphenyl	43 – 116
2-Fluorophenol	21 – 110
Nitrobenzene-d_5	35 – 114
Phenol-d_5	10 – 110
p-Terphenyl-d_{14}	33 – 141

Table 7-13 Internal Standards

Analyte	Accuracy (% R)
Acenaphthene-d_{10}	-50 to +100
Chrysene-d_{12}	-50 to +100
1,4-Dichlorobenzene-d_4	-50 to +100
Naphthalene-d_8	-50 to +100
Perylene-d_{12}	-50 to +100
Phenanthrene-d_{10}	-50 to +100

④ *The Surrogate limits are derived from USEPA CLP OLM 03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of liquid samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits.*

Table 7-14 Extracted Laboratory Control Spikes

Analyte	Accuracy ① (% R)
1,2,4-Trichlorobenzene	39 – 98
1,4-Dichlorobenzene	36 – 97
2,4-Dinitrotoluene	24 – 96
2-Chlorophenol	27 – 123
4-Chloro-3-methylphenol	23 – 97
4-Nitrophenol	10 – 80
Acenaphthene	46 – 118
N-Nitroso-di-n-propylamine	41 – 116
Pentachlorophenol	9 – 103
Phenol	12 – 110
Pyrene	26 – 127

Table 7-15 Pre-Spike Surrogates

Analyte	Accuracy ② (%R)
Benzo(a)Pyrene-d_{12}	50 – 150
Fluoranthene- d_{10}	50 – 150

① *The LCS limits are derived from USEPA CLP OLM03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 - 150% are applied.*

② *The pre-spike Surrogates limits are arbitrary. Air Toxics Ltd. received a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of in-house control limits.*

Table 7-16 Summary of Calibration and QC Procedures SW-846 Modified Method 8270C

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hrs.	SW-846 tuning criteria for Semi-volatiles analysis.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	ICAL criteria in Table 7-10.	Correct problem then repeat Initial Calibration.
Independent Source Calib. Ver. (ISCV)	All analytes – once per Initial Calibration.	At least 90% of the target compounds recoveries must be between 50 – 150%.	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock, immediately after the DFTPP tune check.	**SPCCs:** RF≥0.050 **CCCs:** %D ≤20%; Non-CCC's when CCC compounds are not requested %D ≤20%.	Investigate and correct the problem, up to and including re-calibration if necessary. High bias for one or more compounds associated with non-detects in the samples will not result in re-analysis.
Internal Standards (IS)	As each standard, blank, and sample is being aliquoted.	**For CCVs:** area counts within -50% to +100% from the most recent ICAL. **For blanks, samples and non-CCV QC Checks:** Retention Times within ± 0.33 minutes (20 seconds) and area counts within -50% to +100% of the CCV.	**For CCVs:** Investigate and correct the problem before proceeding with sample analysis. **For blanks:** Inspect the system and re-analyze the blank. **For samples and non-CCV QC:** Re-analyze the samples. If the criteria are not met a second time, dilute sample until IS meet criteria.
Surrogates	With all samples and blanks prior to extraction.	See Table 7-11.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Extracted LCS	With each set of up to 20 extracted samples.	See LCS Criteria in Table 7-13.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Laboratory Blank	With each set of up to 20 extracted samples.	Results less than laboratory RL.	Re-aliquot and re-analyze the extract to confirm the presence of the target compound. If it doesn't confirm, investigate and correct the problem before re-analyzing all the affected samples.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Solvent Blanks	When samples that are extracted together are analyzed on different analytical shifts.	All target compounds below the RL.	Investigate and correct the problem before re-analyzing all the affected samples.
Laboratory Duplicates	10% of the samples.	RPD ≤ 25% for all detections > 5 X's RLs.	Analyze a third time. Report the closest two results and narrate and report the data if the criteria is still not met.

TO-15

8.0 TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS

This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either concentrated using a cryogenic trap and/or concentrated using a hydrophobic multisorbent bed. The hydrophobic multisorbent bed functions as a drying system which removes water from the sample stream prior to analysis by full scan GC/MS. For low level analysis, the sample is focused onto a cryogenic cooled column for analysis by full scan GC/MS.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 8-1 Summary of Method Modifications

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sample Drying System	Nafion Drier.	Multisorbent.	Multisorbent.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blanks and standards **(applies to Low Level analysis only)**	Zero Air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Daily CCV	≤ 30% D.	≤ 30% D.	≤ 30% D with two allowed out to 40% for **QUAD** analysis and four allowed out to 40% for **Low Level** analysis; flag and narrate outliers.
Initial Calibration	≤ 30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % for **QUAD** analysis and 4 compounds allowed out to < 40 % for **Low Level** analysis.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.
Sample collection media.	Summa canister.	Summa canister.	Air Toxics Ltd. recommends use of summa canisters to insure data defensibility, but will report results from Tedlar bags at client request.

Table 8-2 Method TO-14A/TO-15 Analyte List (Standard Compounds)

Analyte	RL (ppbv) TO-15/LL	%RSD TO-15/LL	Acceptance Criteria	
			LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1,2-Trichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1-Dichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1-Dichloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2,4-Trichlorobenzene	2.0/0.5	30%/30%	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichloropropane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,3-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,4-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Benzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Bromomethane	0.5/0.1	30%/30%	70 - 130	≤ 25
Carbon Tetrachloride	0.5/0.1	30%/30%	70 - 130	≤ 25
Chlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloroform	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloromethane	2.0/0.1	30%/30%	70 - 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.5/0.1	30%/30%	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.5/0.1	30%/30%	70 - 130	≤ 25
Dichloromethane	0.5/0.2	30%/30%	70 - 130	≤ 25
Ethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 114	0.5/0.1	30%/30%	70 - 130	≤ 25

Analyte	RL (ppbv) TO-15/LL	%RSD TO-15/LL	Acceptance Criteria	
			LCS (%R)	Precision Limits (Max. RPD)
Freon 12 (Dichlorodifluoromethane)	0.5/0.1	30%/30%	70 - 130	≤25
Hexachlorobutadiene	2.0/0.5	30%/30%	70 - 130	≤25
m,p-Xylene	0.5/0.1	30%/30%	70 - 130	≤25
Methyl Chloroform	0.5/0.1	30%/30%	70 - 130	≤25
o-Xylene	0.5/0.1	30%/30%	70 - 130	≤25
Styrene	0.5/0.1	30%/30%	70 - 130	≤25
Tetrachloroethene	0.5/0.1	30%/30%	70 - 130	≤25
Toluene	0.5/0.1	30%/30%	70 - 130	≤25
trans-1,3-Dichloropropene	0.5/0.1	30%/30%	70 - 130	≤25
Trichloroethene	0.5/0.1	30%/30%	70 - 130	≤25
Vinyl Chloride	0.5/0.1	30%/30%	70 - 130	≤25

Table 8-3 Method TO-14A/TO-15 Analyte List (Non-Standard and Polar Compounds)

Analyte	RL (ppbv) TO-15/LL	%RSD TO-15/LL	Acceptance Criteria	
			LCS (%R)	Precision Limits
1,3-Butadiene	0.5/0.5	30%/30%	60 – 140	≤25
1,4-Dioxane	2.0/0.1	30%/30%	60 – 140	≤25
2-Butanone (Methyl Ethyl Ketone)	0.5/0.1	30%/30%	60 – 140	≤25
2-Hexanone	2.0/0.5	30%/30%	60 – 140	≤25
4-Ethyltoluene	0.5/0.1	30%/30%	60 – 140	≤25
4-Methyl-2-Pentanone (MIBK)	0.5/0.1	30%/30%	60 – 140	≤25
Acetone	2.0/0.5	30%/30%	60 – 140	≤25
Bromodichloromethane	0.5/0.1	30%/30%	60 – 140	≤25
Bromoform	0.5/0.1	30%/30%	60 – 140	≤25
Carbon Disulfide	0.5/0.5	30%/30%	60 – 140	≤25
Cyclohexane	0.5/0.1	30%/30%	60 – 140	≤25
Dibromochloromethane	0.5/0.1	30%/30%	60 – 140	≤25
Ethanol	2.0/0.5	30%/30%	60 – 140	≤25
Heptane	0.5/0.1	30%/30%	60 – 140	≤25
Hexane	0.5/0.1	30%/30%	60 – 140	≤25
Isopropanol	2.0/0.5	30%/30%	60 – 140	≤25
Methyl t-Butyl Ether (MTBE)	0.5/0.1	30%/30%	60 – 140	≤25
Propylene	2.0/0.5	30%/30%	60 – 140	≤25
Tetrahydrofuran	0.5/0.5	30%/30%	60 – 140	≤25
trans-1,2-Dichloroethene	0.5/0.1	30%/30%	60 – 140	≤25
2,2,4-Trimethylpentane	0.5/0.5	30%/30%	60 – 140	≤25
Cumene	0.5/0.1	30%/30%	60 – 140	≤25
Propylbenzene	0.5/0.1	30%/30%	60 – 140	≤25
3-Chloroprene	2.0/0.5	30%/30%	60 – 140	≤25

Analyte	RL (ppbv) TO-15/LL	%RSD TO-15/LL	Acceptance Criteria	
			LCS (%R)	Precision Limits
TPH (Gasoline) or NMOC (Hexane/Heptane)	10	One Point Calibration	NA	≤ 25

Table 8-4 Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 - 140
1,4-Difluorobenzene	60 - 140
Chlorobenzene-d_5	60 - 140

Table 8-5 Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 8-6 Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis.	% RSD ≤ 30 with two compounds allowed out to ≤ 40% RSD (4 allowed out for LL).	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be 70-130%; for 80% of "Non-standard" compounds, recoveries must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Reprepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	%D ≤30% for all compounds with two exceptions not to exceed 40% (four allowed out for LL). Narrate all results outside of compliance. Flag all results outside of compliance with the exception of high bias associated with non-detects.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5 point calibration curve.
Laboratory Blank	After the CCV/LCS.	Results less than the laboratory reporting limit.	Inspect the system and Re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample. If the ISs are within limits in the re-analysis, report

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
		the daily CCV internal standards.	the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.
Laboratory Duplicates	10% of the samples.	RPD ≤25% for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate results.

Sample Handling

The information provided in ATL's Sampling booklet is meant to serve only as general guidelines. In all cases, field sampling personnel are ultimately responsible for having expertise and knowledge in air sampling methodology sufficient to ensure that the defensibility of the data will not be compromised due to deficiencies in field sampling, handling or transportation.

5.3 SAMPLE RECEIVING

Upon arrival at the laboratory, samples are received and inspected following a written Sample Acceptance Policy. The Technical Director ensures that all samples are accepted in accordance with this Policy. The policy establishes specific guidelines for sample acceptance, which are generally accepted practices under EPA, AFCEE, USACE, Navy, and NELAP protocols. When samples do not meet the established guidelines, discrepancies are documented and the client is notified. Samples are noted in the individual work order and discrepancies noted in the Laboratory Narrative portion of the sample report. The Sample Acceptance Policy is provided to field staff with every shipment of containers or media.

5.3.1 Sample Acceptance Policy

Samples received by Air Toxics Ltd. must be relinquished following standard EPA approved guidelines. These include full and complete Chain-of-Custody documentation indicating:

- Unique sample name
- Location, date, and time of collection
- Collector's name
- Preservation type (if applicable)
- Matrix
- Any special remarks

The chain-of-custody form must be filled out in ink and indicate proper preservation and use of sample container specified by the method.

Each sample should be labeled with unique, durable, and indelible identification and must be of adequate volume for the tests requested. Never affix a label directly on a Summa™ canister. A tag is attached to each canister for this purpose.

Proper, full, and complete inspection and documentation will be performed upon laboratory receipt in the following areas:

- evidence of container's physical damage
- status of the container's custody seal
- presence or absence of a chain-of-custody form
- incomplete or incorrect chain-of-custody form
- number of samples
- name of each sample
- sample collection date/time
- sample location
- name of the collector
- preservation type (if applicable)
- sample type (canister, XAD, DNPH etc.)
- sample tag information complete
- temperature (when applicable)
- pressure (canisters)
- presence of unlabelled samples
- presence of mis-labelled samples
- presence of unused media
- method required trip blanks, field blanks, equipment blanks, field duplicates, or field spikes

Any sample discrepancies against the above criteria are documented on the Sample Discrepancy Form (Exhibit 5.3), and communicated to the client via Login Fax within 1 day of sample receipt. The client is contacted by the project manager for discrepancies of a more serious nature, e.g.,

- Chain-of-Custody Record was not received with sample(s).
- Analysis method(s) is(are) not specified.
- Sample(s) received out of holding time.

- Sample container (Tube/VOA vial) was received broken.
- Container for VOA analysis received with headspace.
- Tedlar Bag received leaking.
- Tedlar Bag received flat.
- Tedlar bag / canister received emitting a strong odor (sample cannot be analyzed).

Documentation of client notification is included on the form along with any instructions from the client on how to proceed. Project managers complete this section and return the form to the receiving group to complete the login process. The form is archived in the Work Order folder. Whenever there is any uncertainty of how the laboratory is to proceed or when the desired method is unclear, the receiving staff places the Login process ON HOLD and delivers the Work Order file to a project manager for follow-up. The project manager contacts the client to clarify the situation. Phone calls between the project manager and the client are documented in the Client Services Software. The phone contact and client instructions to resolve the issue are logged into the database and a hardcopy report is placed in the Work Order folder. The folder is then returned to the Receiving team to complete the Login process. Air bills, packing lists, chain-of-custody records, and any other documentation that may accompany the samples are placed in the work order folder.

Laboratory malfunctions occurring during/after sample receipt are documented via the laboratory Corrective Action system. Examples of receiving problems, which would necessitate a Corrective Action Request, include:

- Hold time expired due to laboratory error.
- Canister sample pressurized with wrong type of gas.
- Sample placed On Hold was released in error.
- Sample logged in for incorrect analysis method.
- DANGER tag was not affixed to an odiferous canister sample before sending to the lab.
- Canister was released and cleaned before second analysis method was run.
- Receiving did not affix the multiple analysis tag.
- Canister valve was left open following pressurization. Sample vented to ambient.

5.3.2 The Login E-Mail/Fax

When Login is completed, an email is sent to the client to confirm receipt of samples. If no email address exists a fax is sent for the identical purpose. The Login Email/Fax has five parts:

Page 1 *Cover page with discrepancies noted*
Page 2 *Log-in summary (sample names etc.)*
Page 3 *Reporting template showing referenced method, target compound list, and reporting limits*
Part 4 *Copy of COC*
Part 5 *Media outstanding (if relevant)*

Discrepancies are noted on the cover page using a template of pre-approved statements. The QA Dept. is responsible for maintaining the approved template. Receiving staff electronically copy relevant statements from the template and onto the Email/FAX cover page. Typical statements include:

- NELAC Chapter 5 specifies that a legal Chain of Custody must accompany samples when they arrive at the laboratory. In this case a chain of custody was not received with the samples. The discrepancy was noted in the Login email.
- The COC form was not completed properly. Please note for future reference

that the COC must be signed and dated in order to properly relinquish samples.

- Samples were received past the recommended hold time of _____ hours/days. ATL will proceed with the analysis unless otherwise notified.
- The samples do not have any documentation regarding the date of collection. Unless otherwise notified, the COC relinquish date will be used.
- The Tedlar bag for sample _____ was received flat and could not be analyzed.
- All samples were received at or near ambient pressure yet flow controllers were used. ATL will proceed with the analysis unless otherwise notified.
- Samples were not received at the recommended temperature. ATL will proceed with the analysis unless otherwise notified.

5.3.3 The Work Order Folder

A folder is created during the Login process to hold all relevant documents. The folder is labeled with the unique Work Order number, client name and analysis. One folder for each desired analysis is created so that laboratory analyses can be efficiently handled as separate processes. The folder contains the following receiving documents:

- Login summary sheet with individual field sample names, dates of collection and project reference
- Specific method cited, and a copy of the reporting target compound template for review
- Original COC record, airbill, and any other packing documents
- Original copy of the Sample Discrepancy Report
- Original copy of any CAR Forms
- Copy of the Project Management Project Profile with associated special analysis and reporting requirements
- Copy of any approved Project Requirement tables generated after the bid has been won

The folder is passed to the analytical teams after Login, and follows the same process stream as the samples. All original documents generated during the processing of the samples are filed in this folder. The unique Work Order file makes archival and retrieval of evidentiary and custodial documents easier. The majority of analytical documentation is archived electronically. Documentation that remains in hard copy form includes:

- COC
- Data Review Checklist
- Sample Discrepancy Reports
- Corrective Action Requests
- Scan Packets (run logs, spectral defenses, manual integrations etc.)
- Phone contacts and emails
- Bid Ships/Canister Certifications
- Fed-Ex/UPS air bill/freight bill
- GC/FID screening results

Alternatively, the Work Order folder is placed in a bar coded storage box for long-term storage. Work Order inventory of each box is taken prior to offsite storage and maintained along with the bar code address. A private storage company archives the boxes by barcode and provides one-day retrieval service upon request. Alternatively, work order folders may be scanned onto CD-ROM Media and stored on-site.

5.4 SAMPLE TRACKING PROCEDURES

After samples have been inspected, they are given a unique tracking number and logged into an electronic sample receiving database. The tracking number consists of the year and month plus a sequential Work Order number. As an example, the first set of samples received in July, 2004 would have the format:

0407001

If this set of samples consisted of eight individual samples, then each sample is identified by a consecutive postscript such as:

0407001-01A through 08A

If more than one analysis is requested for the samples, an alphabetic designation is given to each analysis sample set:

0407001A-01A TO-15
0407001B-01A TO-3

Laboratory assigned duplicates are designated using a double postscript such as:

0407001-01AA

A more detailed discussion of the sample receiving function is given in ATL SOP #50. The laboratory processes thousands of samples each month divided into hundreds of individual work orders. An efficient user-friendly database is critical in keeping track of each individual sample, monitoring hold times, monitoring due dates, and scheduling analyses. In addition, most air projects have specific target compound lists, reporting limit requirements, quality assurance requirements, analysis requirements, and data submission requirements. Relevant project information is immediately available as each processing step occurs. The ultimate goal of the ATL sample-processing system is to deliver what the customer wants the first time. Report re-issues and sample re-analyses are monitored and kept to a minimum. In order to meet the quality objective (customer satisfaction), every team member has access to information describing what the customer has requested.

The sample tracking database consists of a variable number of data fields sufficient to store project and sample batch information. The users can then query any field in the database. Each department creates work lists from the database and inputs relevant information (e.g., completion dates, etc.) throughout the day.

The database resides on a secured network server equipped with a daily-automated back up system. Multiple PCs are available to each team in their respective work areas. Access privileges are defined and maintained by the IT team. The database is designed such that work order status can be determined at any point in time. The 'status' field is updated each time the work progresses to a new stage in its processing. Status data include:

- Client Services
- Extractions
- Log-in
- Lab Bins
- Individual Instrument Assignment
- Data Review
- QA
- FAX
- EDD Generation
- Final Report
- Financial Hold
- Filed

Complete documentation of sample processing is maintained in the database. Each team completes relevant portions of the database as work is finished. Selected information includes:

SAMPLE TRACKING FIELDS

- Work Order number
- Client Services contact
- Date received
- Client name
- Project name
- Project ID number
- #Samples
- Date sampled
- #Lab dups
- #Sample holds
- Container type
- Expiration date
- Method specific analysis code
- Date promised
- Rush turn
- 24 hour clock
- Screen done
- Date receiving done
- Receiving analyst initials
- Log-in date
- Log-in analyst initials
- Date analysis done
- Date reported
- Bench analyst initials
- Date of final report
- CVP due date
- Date CVP completed
- Date CVP shipped
- CVP analyst initials
- EDD due date
- EDD completed date
- Date EDD shipped
- EDD analyst initials
- Reissue due date
- Reissue reason
- Time Due

The electronic database is used to document and ensure that analytical hold times, reporting requirements, and project specific QC requirements are met. The database is used by the Client Service Representatives to provide project specific activity reports and status of incomplete work. Users may query the database and easily produce a printed report.

The sample database is the key to efficient information transfer and, as such, is a critical tool to meet the quality objective.

5.5 INTERNAL SAMPLE CUSTODY AND STORAGE PROCEDURES

The chain-of-custody for samples is documented from time of receipt until time of disposal. Internal sample chain-of-custody documentation consists of:

- Storage area logbooks
- Instrument run logs
- Raw analytical data for samples, calibrations and QC checks

The samples are stored in the custody cage, in a secure refrigerator, or in the event of late delivery in the receiving section until the next morning. The receiving staff logs the samples into the Internal Sample or Extractable Sample Tracking Logbook in the storage area.

Samples are tracked in/out of the limited access area by initials, date, and time. All staff members have access to the storage areas and all members are trained on proper custody documentation in the logs. Logbook protocol training is mandatory for all staff. The training and documentation of training is handled by the QA team. The QA team checks the Logbook Review Checksheet monthly to ensure that the analysts have reviewed their logbooks on a timely basis.

5.6 SAMPLE DISPOSAL

Samples are released for disposal upon satisfactory completion of analysis unless prior contractual arrangements have been made. The release of samples is documented in the Internal Sample Tracking Log via a "Released" stamp that includes the date and initials of the person who releases the sample for disposal. Samples are released by the Laboratory Director, Technical Director, Analytical Department Managers and Team

Leaders, Laboratory Scientists or qualified Analysts.

Sample disposal varies based on the sampling media. Whole air samples are vented through a charcoal scrubber, while liquid (i.e., solvent and water) samples are disposed of according to the procedures noted in ATL's Chemical Hygiene Plan.

APPENDIX E-3

Copies of Laboratory Forms/Labels

CompuChem
a division of Liberty Analytical Corp.

CHAIN OF CUSTODY

501 Madison Ave.
Cary, NC 27513
Phone: 919-379-4100 Fax 919-379-4040

Page ____ of ____

Courier
Airbill No.
Sampling Complete? Y or N

Client/Reporting Information	Project Information
Company Name	Project Name
Address	*Sampling Location*
City State Zip	Turnaround time
Project Contact	Batch QC or Project Specific? If Specific, which Sample ID?
Phone #	Are aqueous samples field filtered for metals? Y or N
Sampler's Name	Are high concentrations expected? Y or N? If yes, which ID(s)?

Requested Analysis (include method and bottle type)

Matrices
GW - Ground water
WW - Waste water
SW - Surface water
SO - Soil/Sediment
TB - Trip Blank
RI - Rinsate
WP - Wipe
O - Other

pH / Sample Info (Lab Use)

CompuChem No (Lab Use)	Field ID	Collection		Matrix	# of bottles	Number of Preserved Bottles					
		Date	Time			HCl	NaOH	NHO3	H2SO4	MEOH	Other

Lab Use Only

Sample Unpacked By:
Sample Order Entry By:
Samples Received in Good Condition? Y or N
If no, explain:

Cyanide samples checked for sulfide & chlorine? Y or NA
625 & Phenol samples checked for chlorine? Y or NA
608 samples checked for pH between 5.0-9.0? Y or NA

Comments

Sample Custody

Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:

Subcontact? Y or N If yes, where? | Custody Seal(s) intact? Y or N | On Ice? Y or N | Cooler Temp: °C

White & Yellow copy to lab • Pink copy for customer

P.O. Box 19022
Greenville, SC 29602
1995 Perimeter Rd.
Greenville, SC 29605
(864) 277-1309

QUALITY PRECLEANED
GLASS & PLASTIC
SAMPLING CONTAINERS

PROJECT NAME	TARE WT.
SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	____ GRAB ____ COMPOSITE
ANALYSIS REQUESTED	

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 562-4988 www.essvial.com (800) 233-8425

P.O. Box 19022
Greenville, SC 29602
1995 Perimeter Rd.
Greenville, SC 29605
(864) 277-1309

QUALITY PRECLEANED
GLASS & PLASTIC
SAMPLING CONTAINERS

PROJECT NAME	TARE WT.
SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	____ GRAB ____ COMPOSITE
ANALYSIS REQUESTED	

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 562-4988 www.essvial.com (800) 233-8425

P.O. Box 19022
Greenville, SC 29602
1995 Perimeter Rd.
Greenville, SC 29605
(864) 277-1309

QUALITY PRECLEANED
GLASS & PLASTIC
SAMPLING CONTAINERS

PROJECT NAME	TARE WT.
SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	____ GRAB ____ COMPOSITE
ANALYSIS REQUESTED	

CUSTODY SEAL

DATE _____

SIGNATURE _____

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

@ AIR TOXICS LTD.
AN ENVIRONMENTAL ANALYTICAL LABORATORY

CHAIN-OF-CUSTODY RECORD

Sample Transportation Notice
Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630-4719
(916) 985-1000 FAX: (916) 985-1020

Contact Person _____
Company _____
Address _____ City _____ State ___ Zip _____
Phone _____ FAX _____
Collected By: Signature _____

Project info:
P.O. # _____
Project # _____
Project Name _____

Turn Around Time:
☐ Normal
☐ Rush _____ Specify

Lab I.D.	Field Sample I.D.	Date & Time	Analyses Requested	Canister Pressure / Vacuum Initial	Final	Receipt

Relinquished By: (Signature) Date/Time — Received By: (Signature) Date/Time
Relinquished By: (Signature) Date/Time — Received By: (Signature) Date/Time
Relinquished By: (Signature) Date/Time — Received By: (Signature) Date/Time

Notes:

Lab Use Only

Shipper Name	Air Bill #	Opened By:	Temp. (°C)	Condition	Custody Seals Intact?	Work Order #
					Yes No None	

Form 1293 rev. 09

CUSTODY SEAL

Date _____

Signature _____

Nalge Nunc International

I-CHEM® Brand Products

90009

@ AIR TOXICS LTD.

FIELD SAMPLE I.D. #: _____

CLIENT NAME: _____

PROJECT: _____

SAMPLERS NAME: _____

DATE: _____ **TIME:** _____

CANISTER #: _____

COMMENTS: _____

ANALYSES: _____

INITIAL VACUUM "Hg	FINAL VACUUM/PRESSURE "Hg/psi	DATE	INIT.

LAB SAMPLE I.D. #: _____

DATE RECEIVED: _____ **EXPIRATION DATE:** _____

DATE OF DISPOSAL: _____